General information about the SEM

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General information about the SEM

- We have a LEO Gemini 1530 with a Thermo Scientific UltraDry Silicon Drift Detector (SDD). The SEM was manufactured in Oberkochen, Germany by LEO in 2001. LEO has been bought by Carl Zeiss. The X-ray detector was manufactured by Thermo Scientific in Madison, Wisconsin, U.S.A in 2009.
- The lightest detectable element is beryllium (demands special preparation) for the Thermo Scientific UltraDry detector. **Although the X-ray detector can detect elements from beryllium and on, it is worth noting that the elements from beryllium to neon in a quantitative analysis will give results that have higher uncertainty than heavier elements.**
- The SEM is equipped with a SE (secondary electron), a BSE (backscattered electron) and an In-Lens detector.
- **The magnification of the image corresponds to a Polaroid 545 print with the image size of 8.9x11.4 cm. This should be mentioned in articles.**
- All liquids (water and oil based) will evaporate in the SEMs as the vacuum will be about 10^{-6} mbar.

General information about the coaters

- We have an Emscope TB 500 Temcarb for coating samples with a layer of carbon thru evaporation and an International Scientific Instruments E5000 sputter coater for coating with the metals: gold, copper and palladium.

Before the SEM analysis

Points worth remembering

- We're moving to a web based booking service OpenIRIS, at some point in the future bookings will only be possible using it. The website can be found here: https://openiris.io/ The move hasn't been completed yet but it is worth keeping in mind when reading the second point. If you've got an account to OpenIRIS, feel free to book using it. Contact me if you need help.
- Please contact the SEM operator the moment you know you'll have/get an urgent sample for the SEM, even if you don't know the exact time. A scheduled time is easier to swap with another if the date changes.
- The SEM cannot analyse a sample that is gaseous or liquid.
- The sample should not be wet or contain moisture. This is especially true if the sample is porous, as it might take a VERY long time to pump the vacuum for the SEM. Please dry the sample beforehand!
- If possible, bring the sample to the SEM laboratory a day before your scheduled time.
- Think about what information you want to get from your sample **before** you come to the SEM laboratory. Do you just want to get images of your sample or maybe also elemental analysis?
- Do you want to replicate images seen in a report? Please bring the report with you!
- Is the sample the start of a series of samples/sample belonging to a series? Please tell the SEM operator so that the images and/or analysis will be done in the same way for all samples.
- If you need images for a poster or larger resolutions images for other work, please inform the SEM operator about it.
- Please contact the SEM operator if you have any questions.

How to handle and prepare a sample

Marking a sample and project information

- ***Always mark the sample if it's biohazardous, toxic, poisonous, radioactive, noxious or otherwise harmful!***
- ***Always ask the SEM operator <u>before</u> you come if you can bring a sample like the one described above to the laboratory!***
- Name your sample clearly and unambiguously, also on the sample itself if possible. The name should be as clear as if it was from a printer. Do this even if you'll be present during the microscopy as a sample might be mistaken for another during preparation/coating.
- Samples with more than one analysable surface, i.e. paper or metal plates, should have markings indicating which surface to analyse.
- You can scratch or etch the sample name into the sample, if possible. Another way is to include a piece of paper with the name on it into the resin if you're mounting a sample.
- One way to make marking the samples easier is to number them from 1 onwards and then write the names in a list with the corresponding number.
- If there is a sample list in Excel or on paper, the names on the samples should match those on the list exactly.
- Always write down on an accompanying piece of paper:
    - your name and institution/company
    - contact information
    - project name and number
    - booked date and time/deadline(s)
    - want to present during microscopy

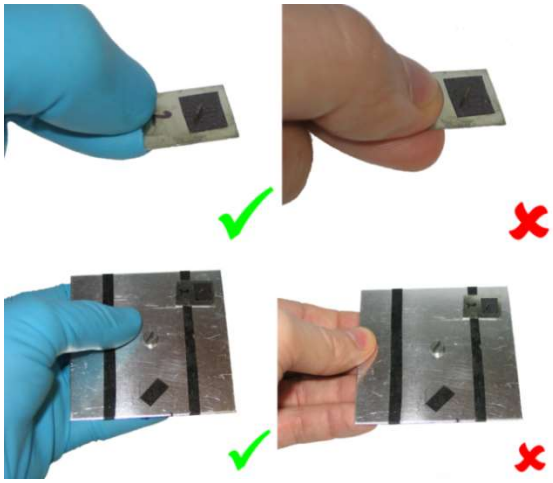
  and leave it with the sample.

Black bag test

- A good way to determine if you've marked the sample properly is to imagine that you throw all the samples in a black bag, shake it around and ask a random person to pick out a sample out of the bag and identify it.
- If the person is able to identify the sample and what surface is to be analysed just by looking at it, you've passed!

Keep it clean and dry

- **Oil and fatty acids (your fingerprints) are SEM killers!** The oil will evaporate in the high vacuum of the SEM and attach itself to the detectors and electronics, degrading their performance over time.
- Use gloves when touching your sample and anything that will go into the SEM. Ethanol can be used to clean away accidental fingerprints. Rinse with distilled water afterwards, if the sample allows it.



- Wash an oily sample thoroughly with for example acetone or petroleum ether. Remember that oil might be in cracks in the sample. Use vacuum to get oil out of cracks and then wash the sample again. Repeat as many times as necessary.
- Clean your sample well from dust, chips from grinding and dirt. Try to use compressed air first. If that doesn't work, ethanol or other solvents can be used, but please rinse it with distilled water afterwards, if the sample allows it, and dry with compressed air.
- Dry your sample thoroughly before bringing it to me. If the sample is porous and has been in a liquid, please dry it overnight in a low-temperature oven or incubator, if possible.
- Don't try to dry the sample in the coater or the SEM as it might have an adverse effect on the sample. It might cause cracking of the surface of the sample.

Amount, shape and size

- Please inform the SEM operator if the sample you bring is all you have of it and you need it back. It's better to only bring a smaller amount of the sample (usually powders) if you've got a lot of it. Also tell the operator if the sample needs to be analysed somewhere else afterwards as a coating on the sample might prevent a proper analysis on another analysis machine.
- The Leo 1530 Gemini can take a sample with the dimensions of 75x75x35 mm. The SEM can be configured to take a slightly taller sample.

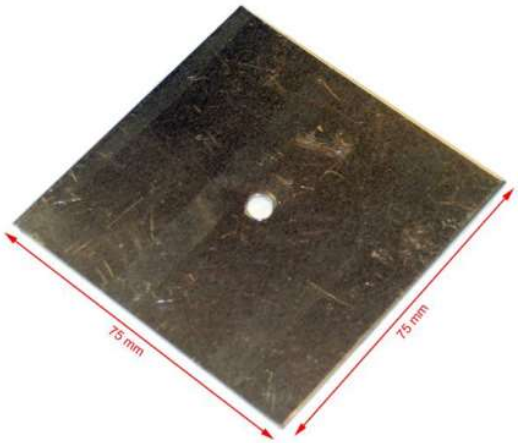
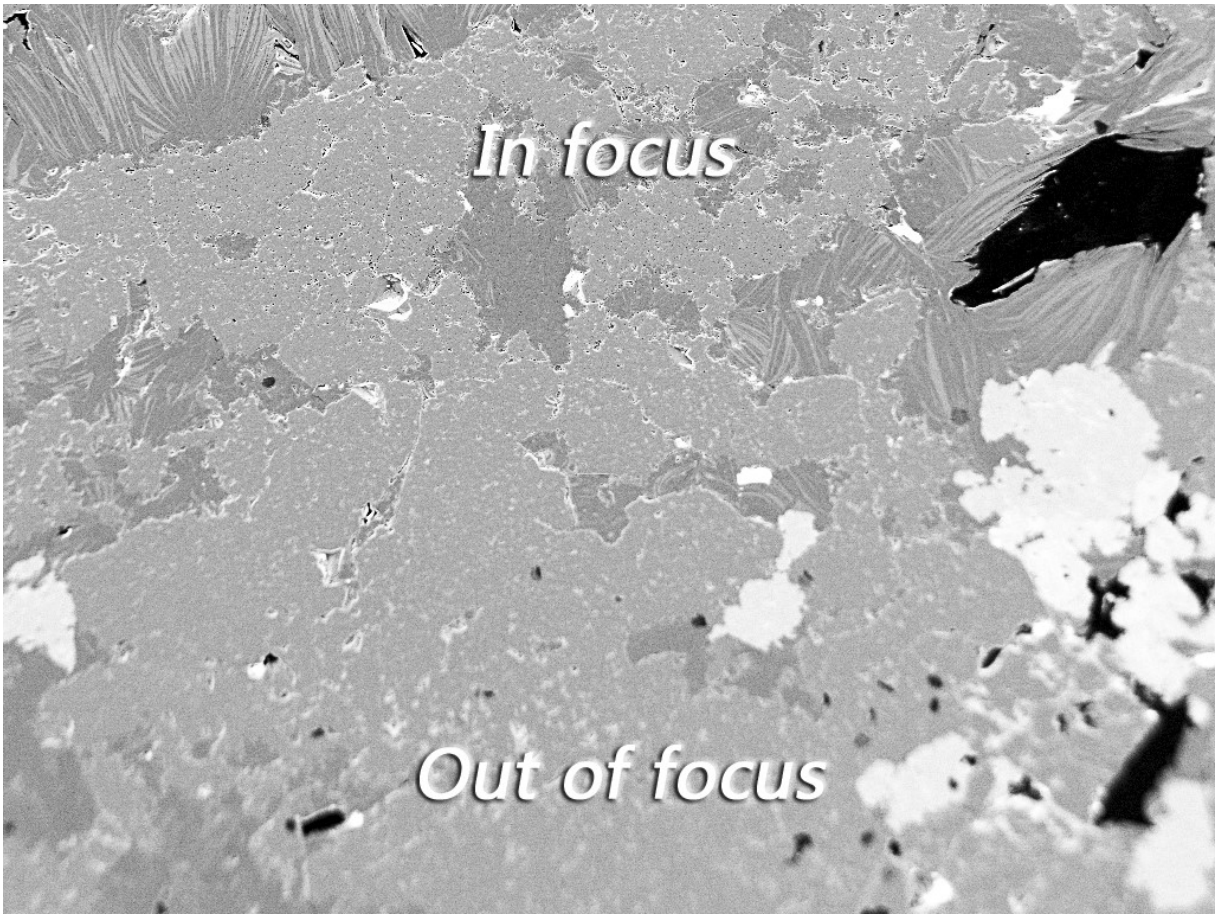


Figure 1. Specimen plate from the SEM to the left, notice the hole for the screw.

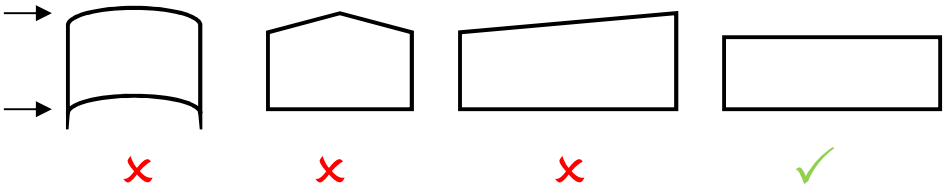
- Try to get the height of the sample as low as possible.
- If you've got more than one sample, try to get the height to be the same across all samples.



- Not having an even sample where both sides are parallel might give you images like the one above where one side is in focus and the other is out of focus.

Mounting a sample in resin

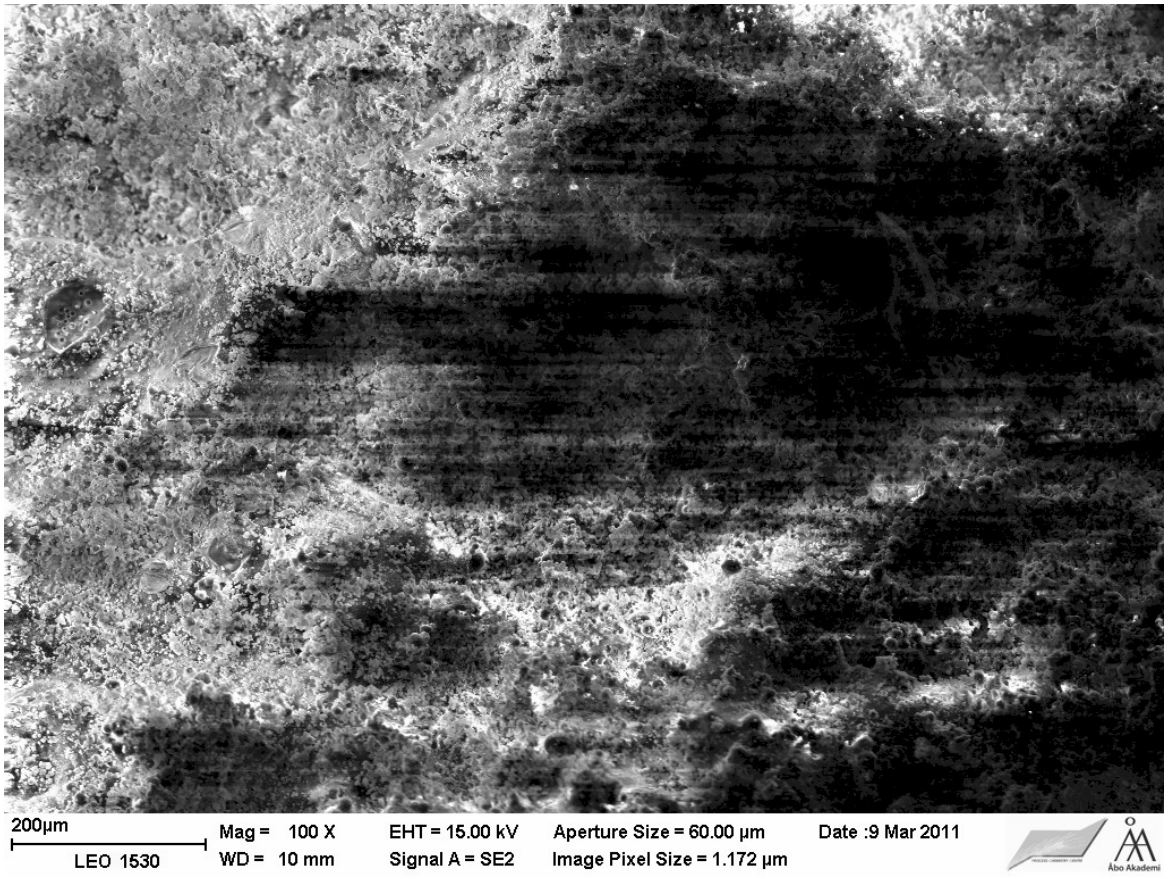
- Try to get both sides of a sample parallel if at all possible, remove pastes or other residues from the bottom as well, it will speed up the microscopy and you'll get better quality results. When you mount your sample in resin, please remember to do a rough grind of the opposite side so that it's flat. It will make it easier to stick it to the specimen plate.



- If you want to study a cross section of a powder or small particles, mount it in resin, i.e. Epofix, and then grind it and polish it until you get the cross section of the sample.
- Don't bring a solid sample which contains loose dust or powder. Mount it in resin if you can't get it dust free, if possible.
- If you grind or cut a sample mounted in resin and find that the crossection has voids or cracks due to the mounting or the sample geometry, please put a layer of resin on top of the sample and then put the sample in vacuum to get it to get in to all the cracks and voids. Regrind the sample after it has cured. This will give a much better surface to analyse and avoid liquids trapped in the sample coming up to the surface and contaminating it.

Coating

- Tell the SEM operator if your sample is electrically conductive and/or sensitive to heat.
- If the sample isn't electrically conductive by itself it has to be coated to make it conductive. Coating alternatives are carbon (most used), gold, palladium and copper. The coating will be present in an elemental analysis.
- Sometimes a sample that has been coated will still charge making it difficult to get good pictures. This might happen with powders, zeolites being a prime example, and a sample that contains oil or other fouling elements. A bad coating might also be the reason why the sample is charging.



- A badly conducting sample might give you an image like the one above.

For elemental analysis

- The best elemental analyses will be had from a sample that is self-conducting, homogenous and has an even surface.
- An analysis of an irregularly shaped sample is also possible.
- If you want a bulk elemental analysis from a powder, please grind it down into a fine powder and press it into a pellet, if possible. The results you get will be a lot better than an analysis from a big grained powder.
- Keep in mind when doing an elemental analysis that you might get carbon, gold, palladium, aluminium and/or copper from the coating/sample holder/mounting. If you want to analyse these elements, let the SEM operator know of it beforehand. The coating, if any, will influence the EDXA results.

Special considerations

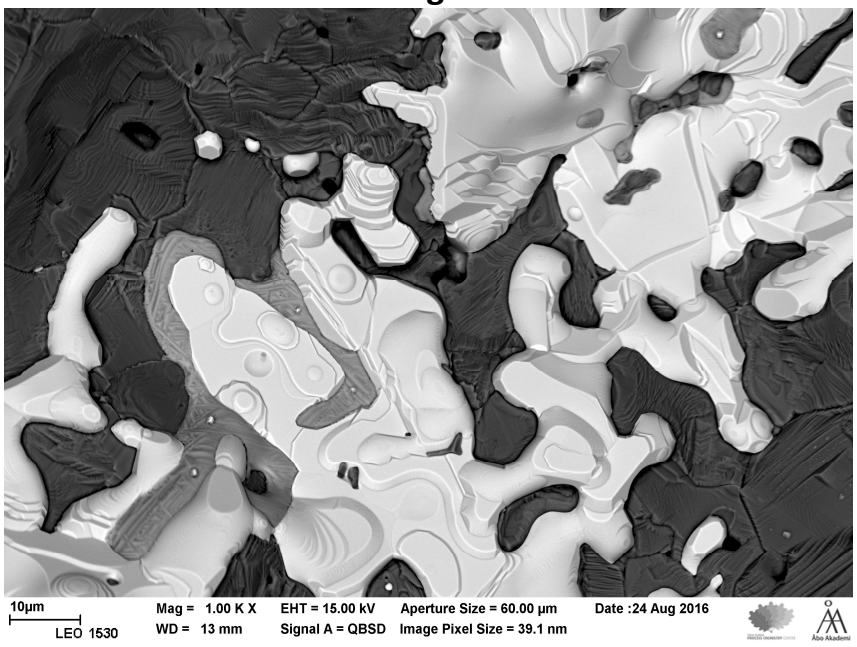
- Sometime a high resolution photograph of the sample is a good thing to have if you want to analyse differently coloured areas. The SEM only "sees" grey but with a good quality photograph, areas can be matched up, with the help of shapes, between the photograph and the SEM image.

- The SEM can’t see through glass like an optical microscope. If it isn’t on the surface of the glass, the SEM won’t see it.

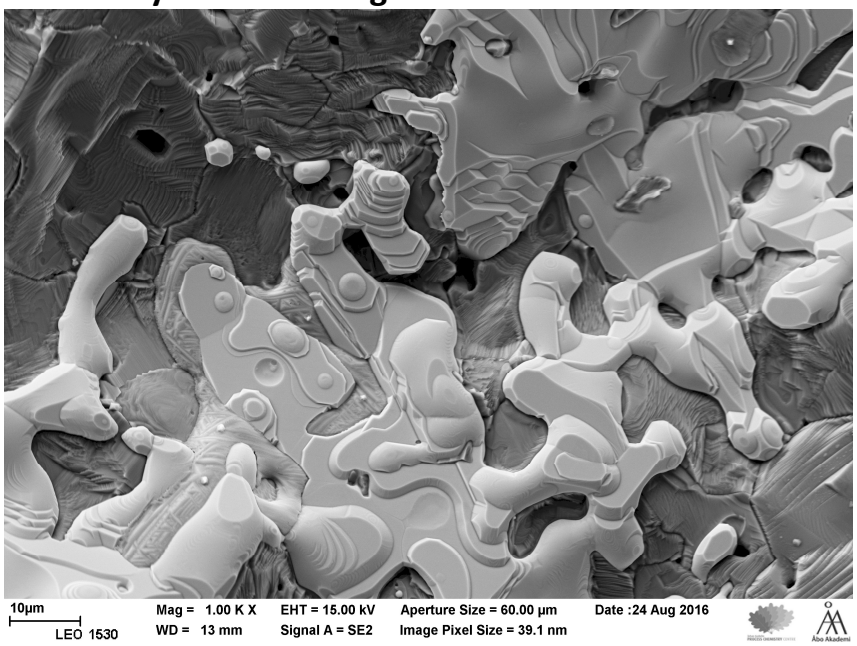
Information available from the SEM

Images

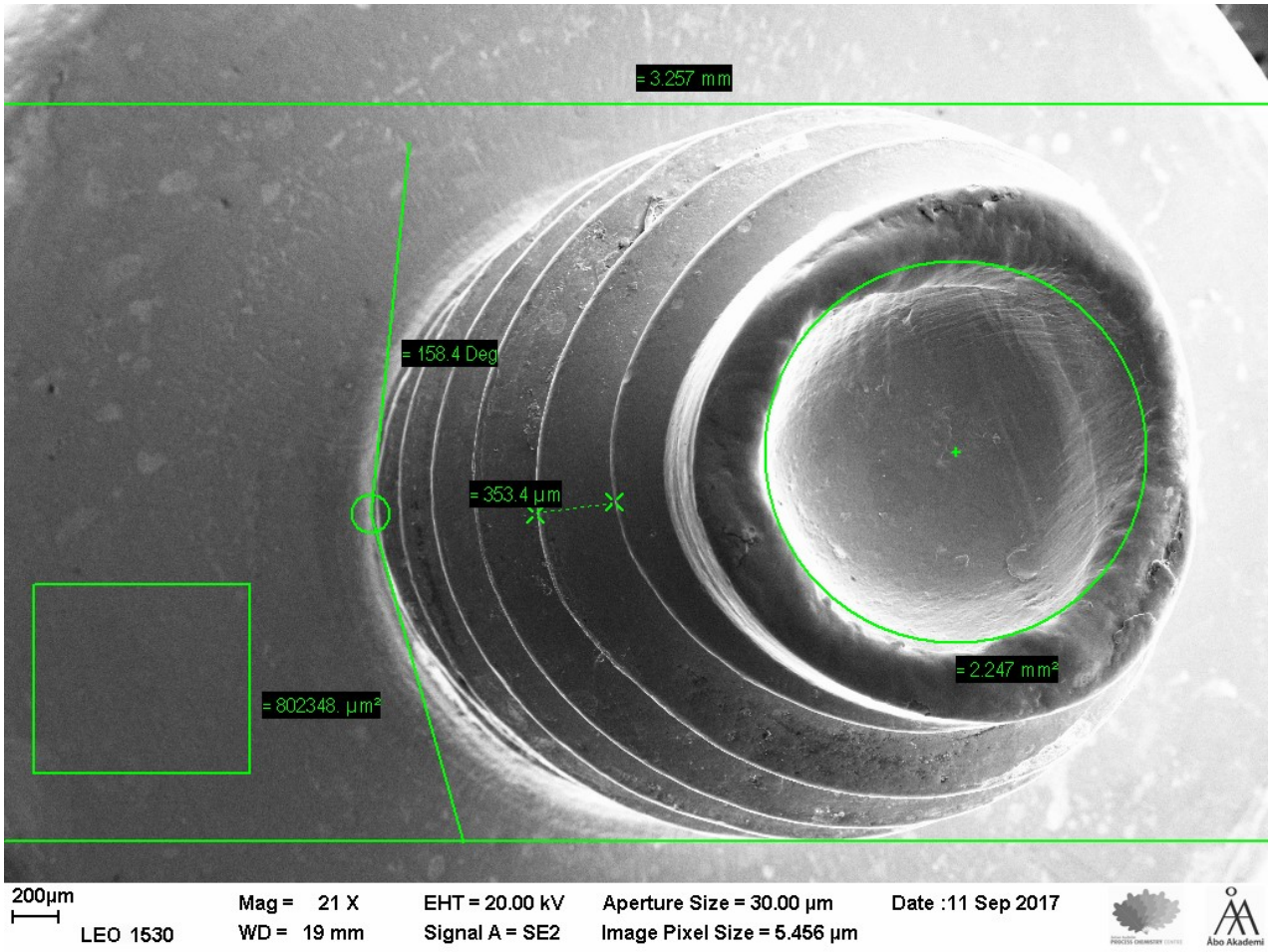
Backscattered electron images



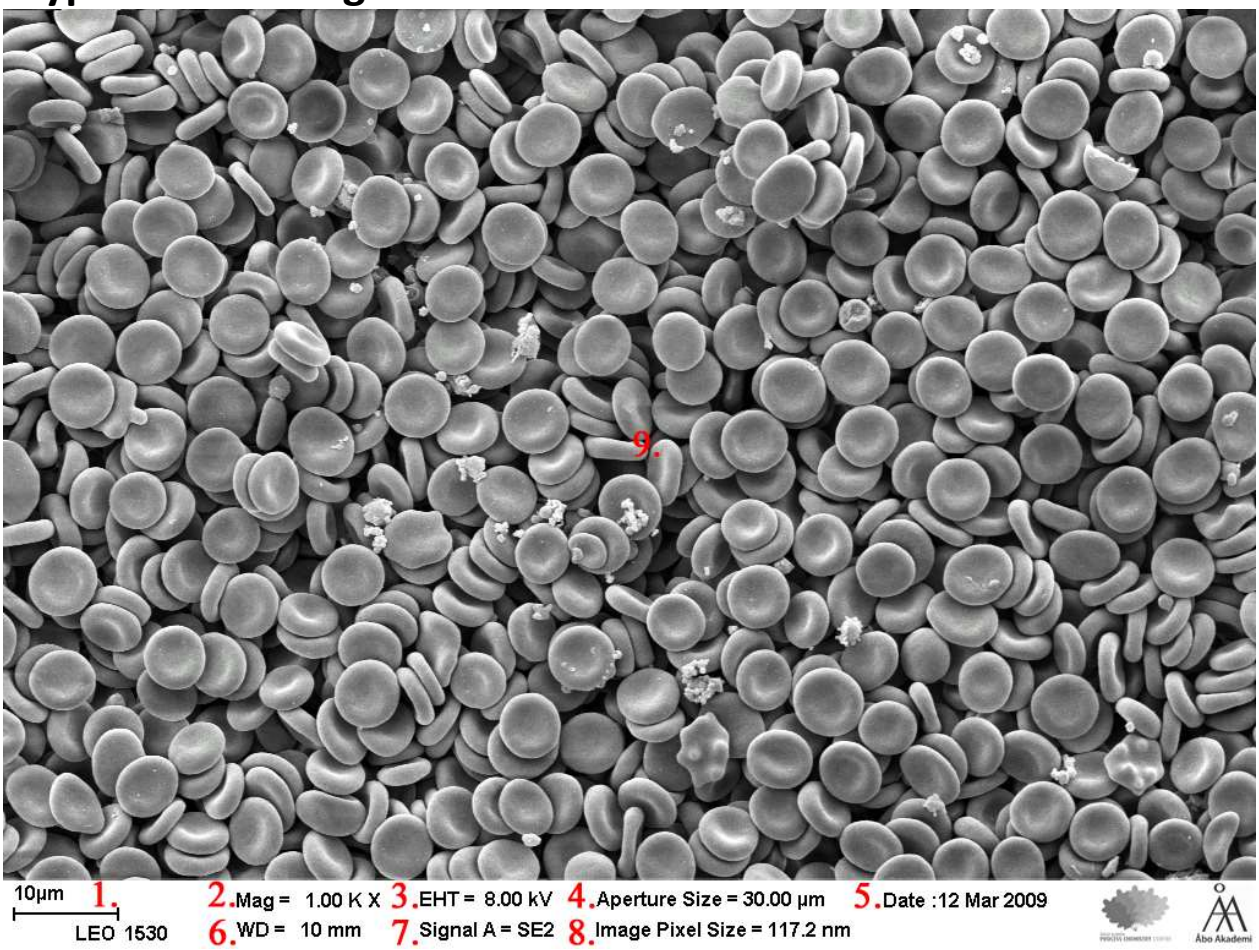
Secondary electron images



Measurements



A typical SEM image with databar

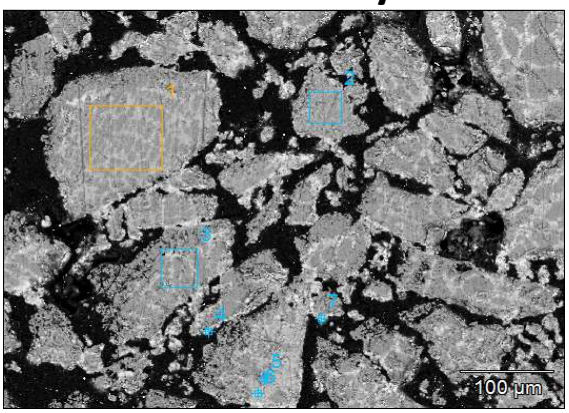


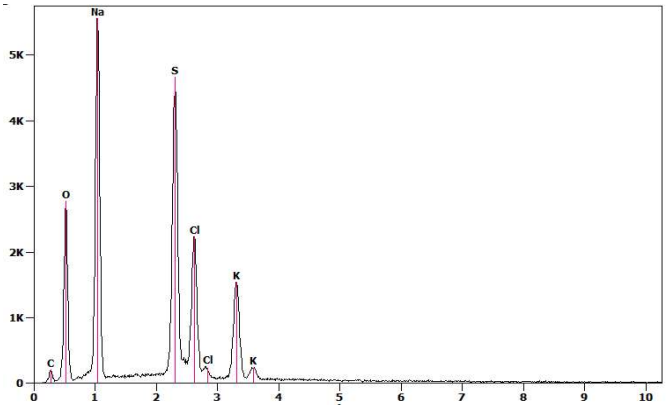
1. The scale bar. The length of the line is in this case 10 µm. “LEO 1530” beneath the line is the name of the SEM.
2. The magnification. In this case the magnification is 1000 times. The K denotes 1000. The magnification corresponds to a Polaroid 545 print with the image size of 8.9x11.4 cm.
3. The acceleration voltage. In this case 8000 V. The higher the voltage the higher the energy of the electrons hitting the sample. Maximum is 30,000 V.
4. The aperture size. The larger the aperture the more electrons hit the sample. Can be 7.5, 10, 20, 30, 60 and 120 µm.
5. The date the image was taken.
6. The working distance. Distance between the pole piece and the sample.
7. The detector in use. SE2 is the secondary electron detector, InLens stands for the in lens detector and QBSD is the backscatter electron detector.
8. The image pixel size. In this case the side of one pixel is 117.2 nm.
9. An image of red blood cells.

All this information and more are is also stored in the TIFF image.

Information available from the EDS

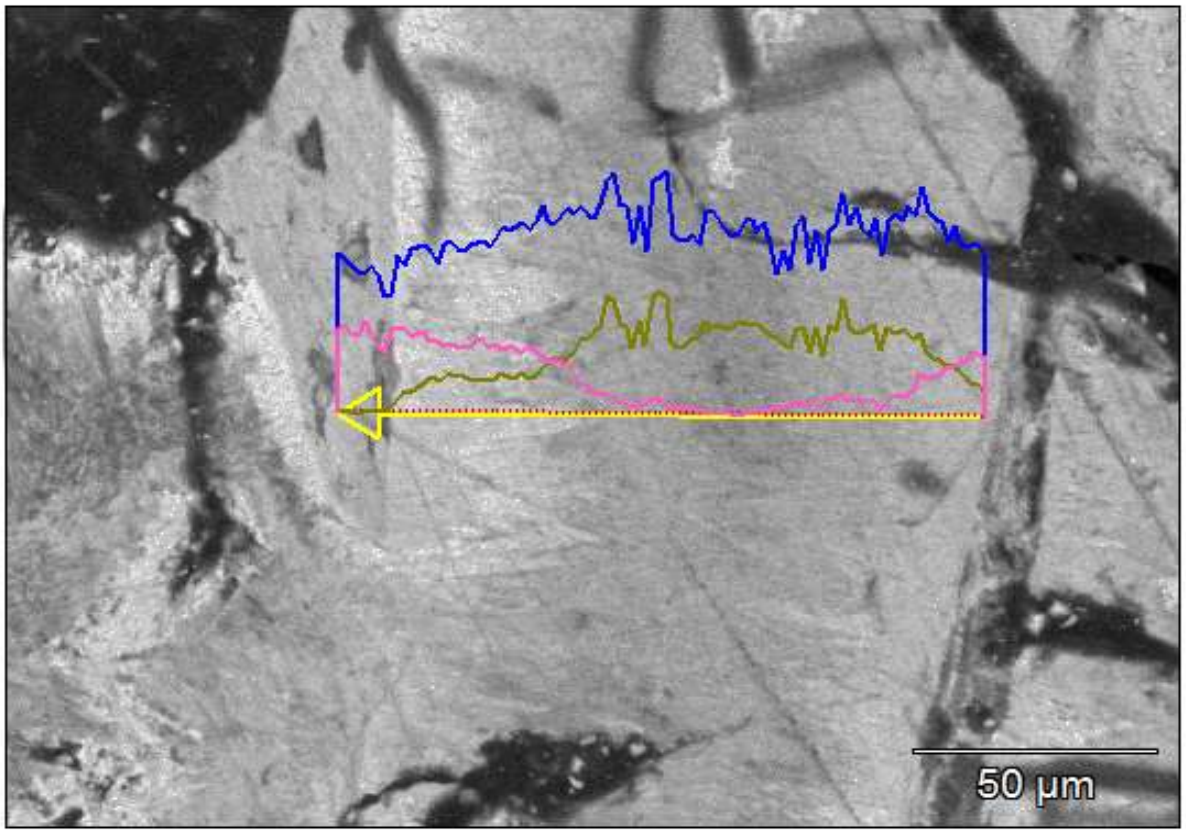
Point and area analysis

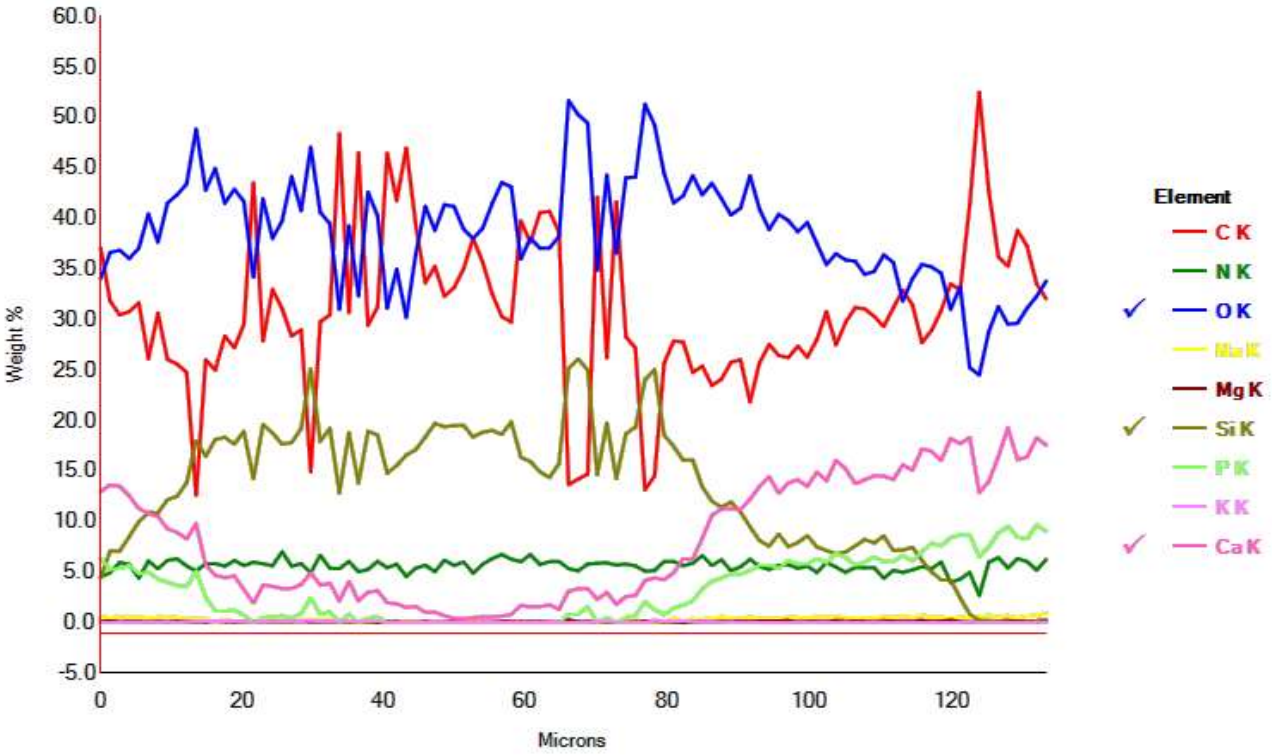




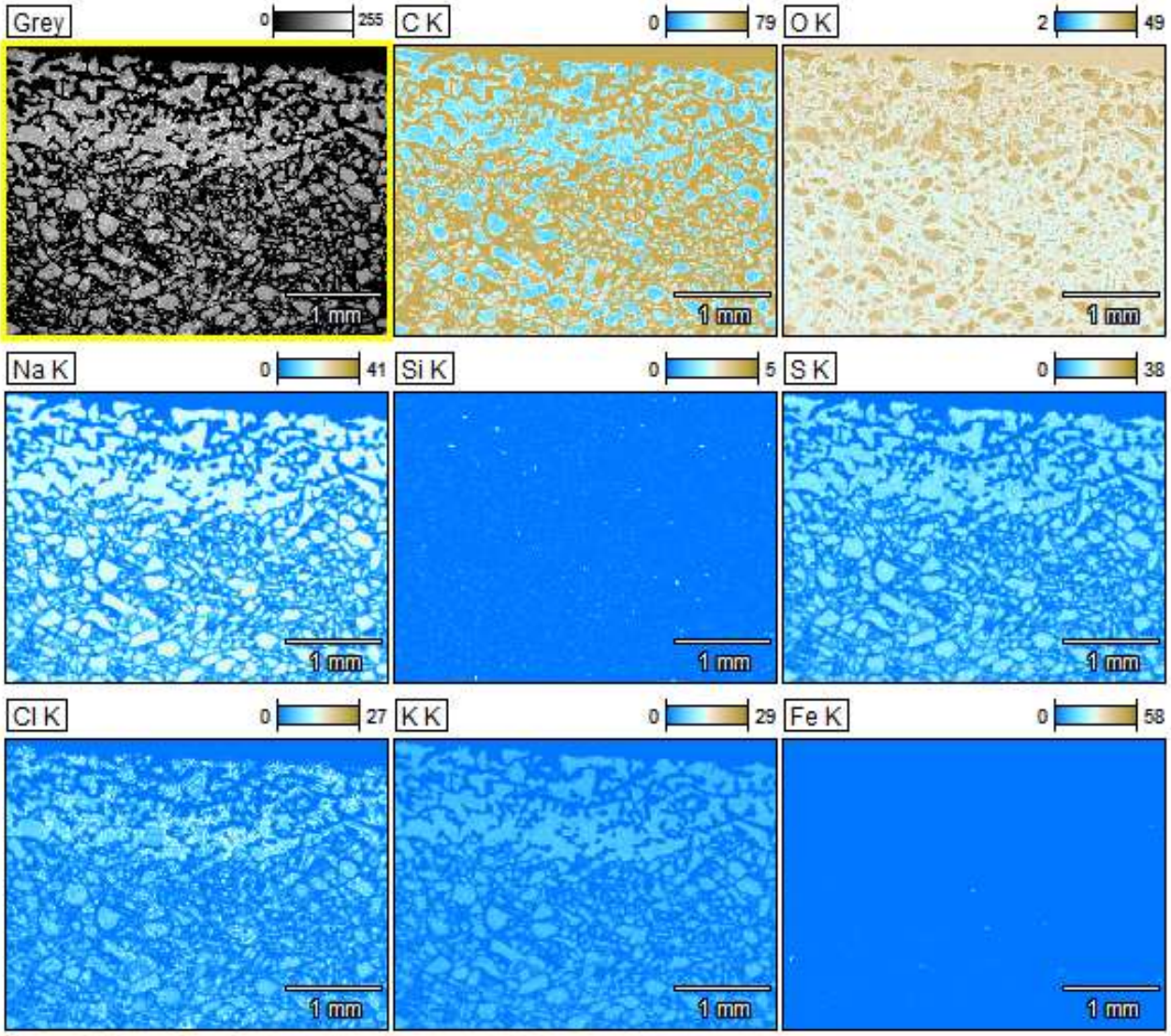
Element	10CrMo 10K 10Cl 400-780oC 4h 200x (4)_pt1						
	Line Type	Weight %	Weight % err	Atom %	Atom % err	Chemical Formula	Compound %
O K (C)	K	36.95	0.36	50.87	0.49	O	36.95
Na K	K	27.64	0.17	26.48	0.16	Na	27.64
S K	K	16.91	0.13	11.62	0.09	S	16.91
Cl K	K	10.56	0.08	6.56	0.05	Cl	10.56
K K	K	7.95	0.10	4.48	0.06	K	7.95
		100.00		100.00			100.00

Linescan analysis

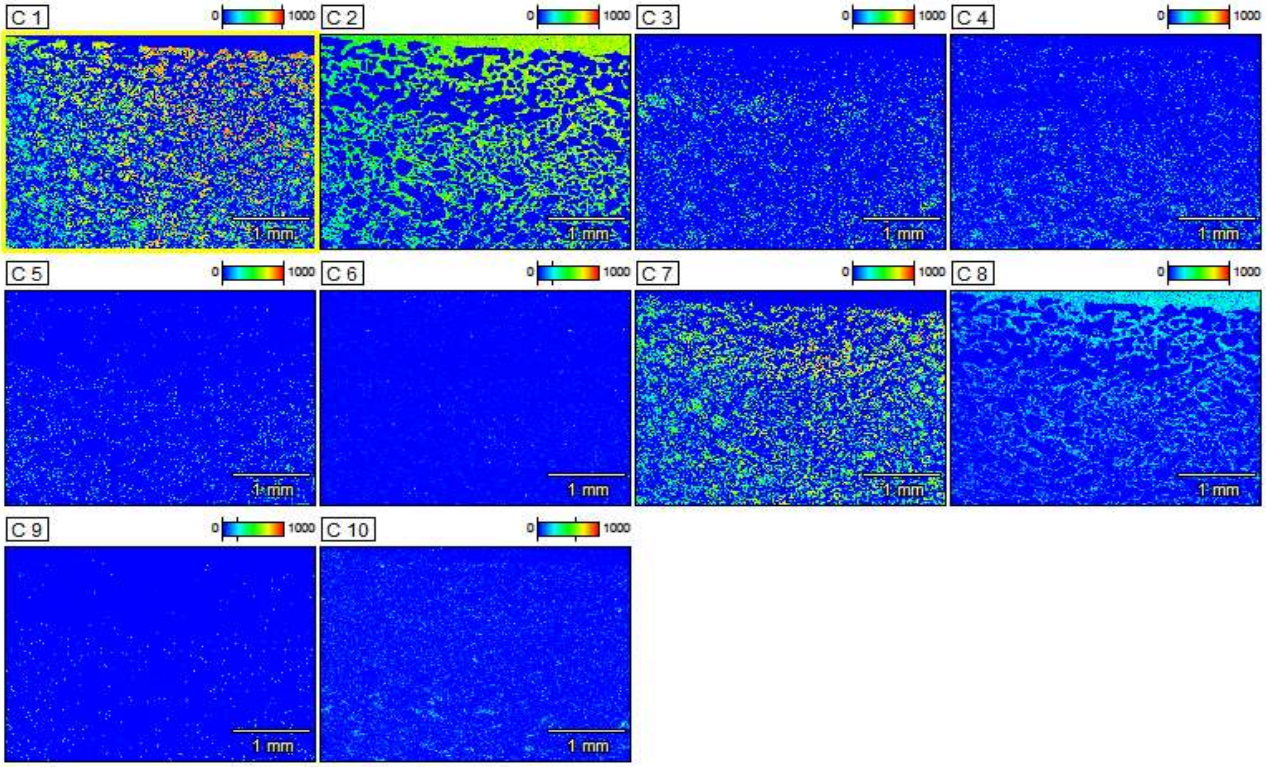




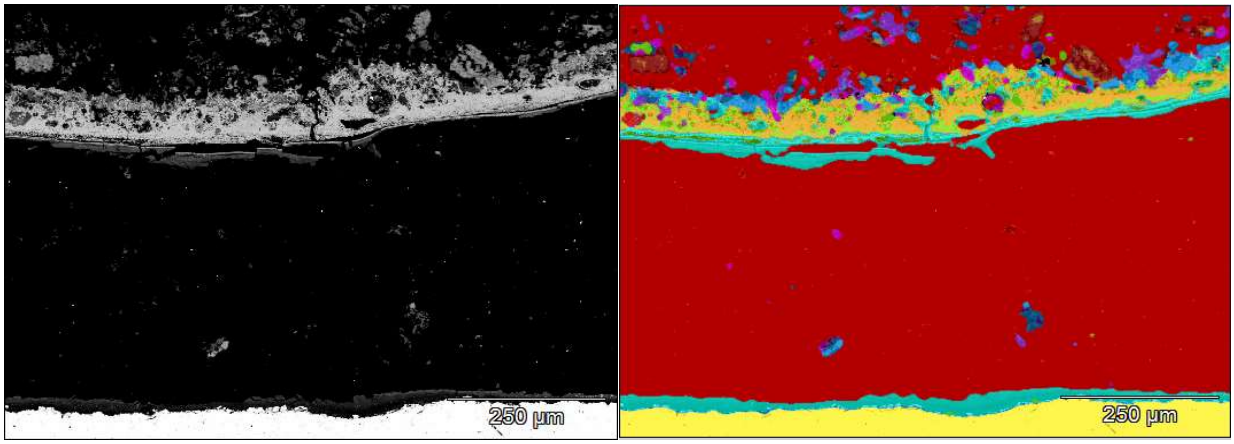
X-ray maps

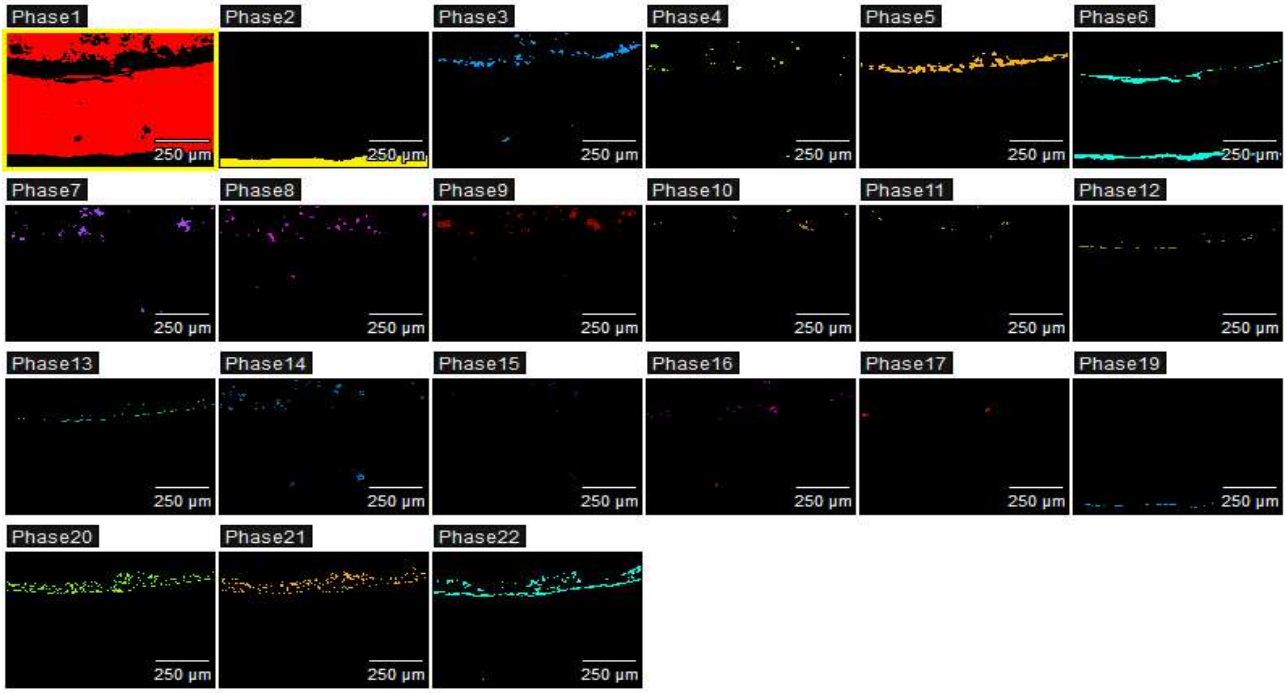

Grey
0
255
1 mm
C K
0
79
1 mm
O K
2
49
1 mm
Na K
0
41
1 mm
Si K
0
5
1 mm
S K
0
38
1 mm
Cl K
0
27
1 mm
K K
0
29
1 mm
Fe K
0
58
1 mm

Compass maps from X-ray maps (needle in the hay stack)

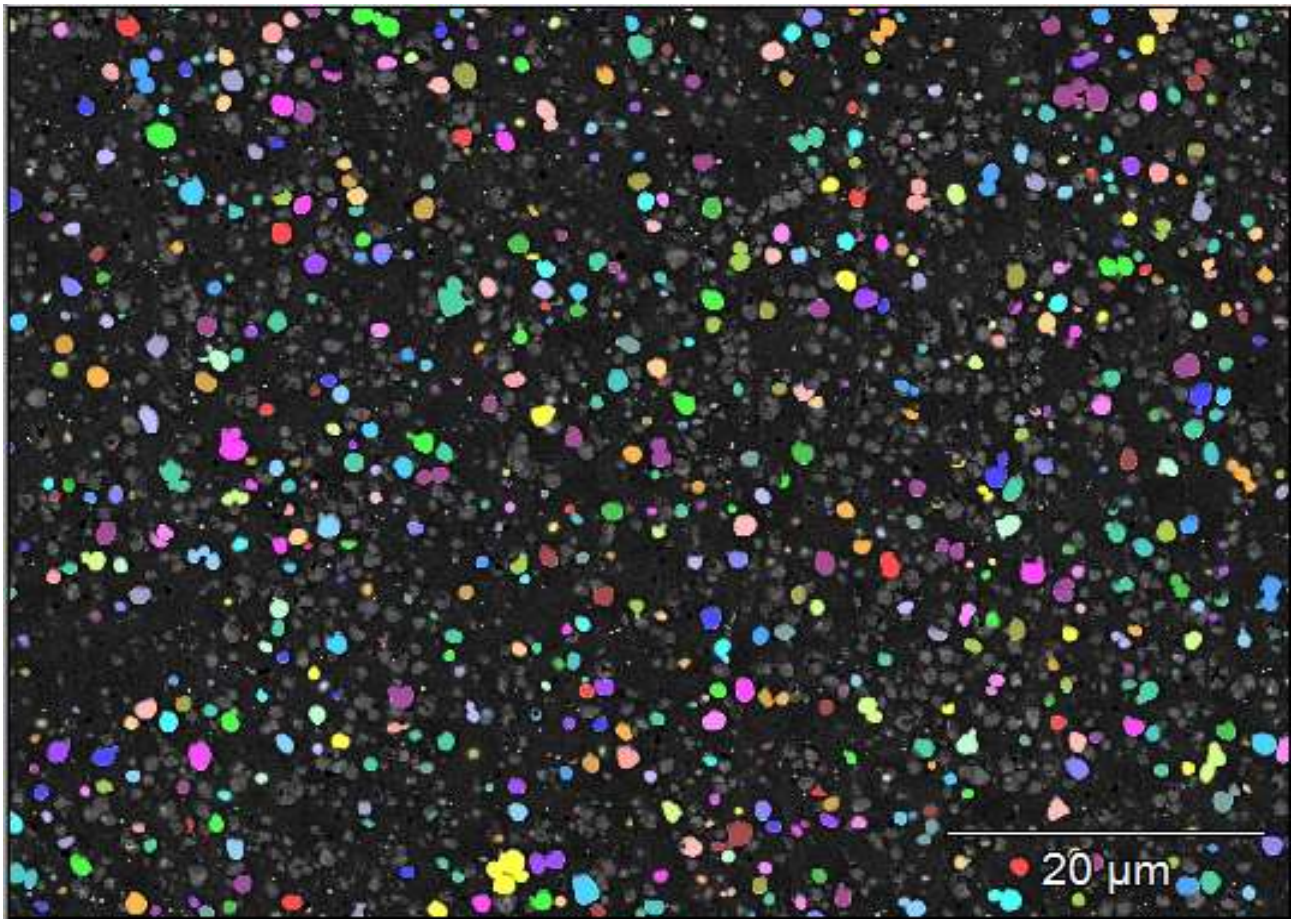


Phase maps from X-ray maps





Particle analysis with elemental characterization



Particles | Frames | Particle Count Summary

Total Particles	Frame #	Particle #	Area	Perimeter	Circularity	X COFM	Y COFM	X Ref	Y Ref	X Feret	Y Feret	X Min	X M
1700	2	845	0.54	2.59	1.0	157	763	157	763	0.94	0.71	151	16
1701	2	846	0.08	0.98	1.0	213	766	213	766	0.47	0.24	210	21
1702	2	847	0.43	3.03	1.7	279	762	279	762	0.79	0.94	274	28
1703	2	848	1.01	3.61	1.0	342	762	342	762	1.26	1.10	334	34
1704	2	849	0.67	2.92	1.0	470	763	470	763	1.02	0.86	464	47
1705	2	850	0.25	1.70	0.9	505	765	505	765	0.63	0.47	501	50
1706	2	851	0.10	1.07	0.9	593	766	593	766	0.47	0.31	590	59
1707	2	852	0.25	2.02	1.3	644	766	644	766	0.94	0.39	639	65
1708	2	853	0.06	0.69	0.6	695	766	695	766	0.31	0.24	693	69
1709	2	854	1.41	5.00	1.4	723	760	723	760	1.73	1.26	712	73
1710	2	855	0.04	0.68	0.9	783	767	783	767	0.39	0.16	781	78
Mean	1.500	428.000	0.568	2.443	0.995	517.821	379.223	517.821	379.223	0.786	0.797	513.284	522.2
Median	1.000	428.000	0.390	2.191	0.974	518.682	379.763	518.682	379.763	0.785	0.710	513.000	524.0
Std Dev	0.500	246.889	0.569	1.581	0.318	292.763	225.871	292.763	225.871	0.434	0.467	292.778	292.8

A typical EDX analysis report in Microsoft Word

Project: 2015-10-16

1. Sample 1

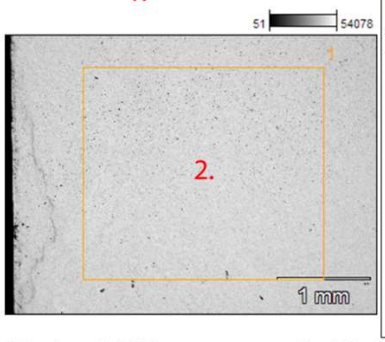
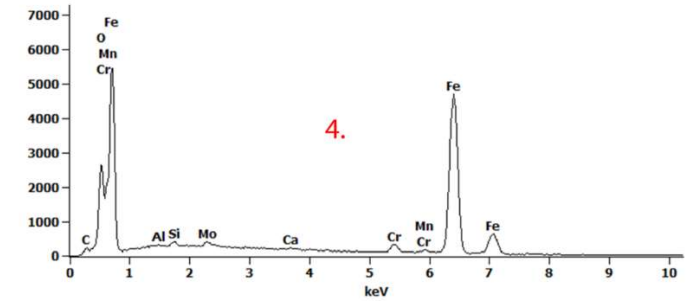


Image Name: Sample 1
Image Resolution: 1024 by 768
Image Pixel Size: 3.93 µm
Acc. Voltage: 15.0 kV
Magnification: 30

3.

Full scale counts: 5447 Sample 1_pt1



Weight %

5.

	O-K	Al-K	Si-K	Ca-K	Cr-K	Mn-K	Fe-K	Mo-L
Sample 1_pt1	5.15	0.17	0.41	0.19	2.10	0.72	90.42	0.84

Weight % Error (+/- 1 Sigma)

	O-K	Al-K	Si-K	Ca-K	Cr-K	Mn-K	Fe-K	Mo-L
Sample 1_pt1	±0.16	±0.03	±0.03	±0.04	±0.13	±0.11	±0.57	±0.12

Atom %

6.

	O-K	Al-K	Si-K	Ca-K	Cr-K	Mn-K	Fe-K	Mo-L
Sample 1_pt1	15.87	0.30	0.72	0.23	1.99	0.65	79.81	0.43

Atom % Error (+/- 1 Sigma)

	O-K	Al-K	Si-K	Ca-K	Cr-K	Mn-K	Fe-K	Mo-L
Sample 1_pt1	±0.50	±0.06	±0.05	±0.05	±0.12	±0.10	±0.51	±0.06

Formula

7.

	O-K	Al-K	Si-K	Ca-K	Cr-K	Mn-K	Fe-K	Mo-L
Sample 1_pt1	O	Al	Si	Ca	Cr	Mn	Fe	Mo

Compound %

	O	Al	Si	Ca	Cr	Mn	Fe	Mo
Sample 1_pt1	5.15	0.17	0.41	0.19	2.10	0.72	90.42	0.84

1. The name of the sample.
2. Image where the area(s) analysed is/are marked.
3. Information about the image and analysis.
4. The EDXA spectrum as a graph.
5. The calculated elements in weight %.
6. The calculated elements in atom %.
7. The calculated elements are sometimes represented as oxides or other compounds here. If not it will show the calculated elements in weight %.

The –K, –L or –M after the element tells which electron shell is used when calculating the weight-%. Usually C is left out of the quantitative analysis if the sample has been carbon coated, but this is up to you.

The results

You can get the results on/via

- CD
- DVD
- USB memory stick
- a networked drive (login information may be required)
- paper
- email
- FUNET Filesender

Points worth remembering

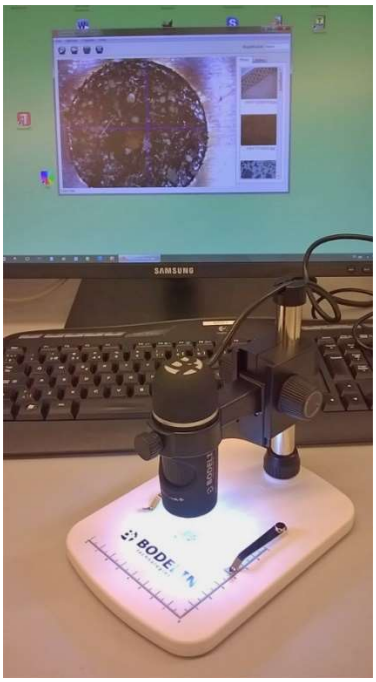
- If there are errors in the naming of the files or folders, ***please tell the SEM operator***, so that they can be corrected on the SEM computers. That way you can have a cup of coffee while the errors are fixed for you.
- It's possible to remove and force elements into the elemental analysis. Results can also be calculated as oxides, useful for people working with glass.
- If you're uncertain about the result you've got, you can always ask the SEM operator about it.

Other services

Vacuum storage

If your sample is sensitive to air or moisture, you can store it in the vacuum chamber until SEM analysis. The vacuum chamber isn't intended for long term storage of samples.

USB microscope



A basic USB microscope is available to take images of your sample before SEM analysis to be used, for example, as a map or colour reference.

Useful programs

Here I've listed some programs that might be useful when you receive or analyse your SEM images.

WinRAR http://www.rarlab.com/download.htm
This is a file archiver/packer that can handle both zip- and rar-files. If images are sent via email they'll be packed and automatically split to minimize the number of emails that are sent. Please get this program if you want results sent via email.

ImageJ http://rsbweb.nih.gov/ij/
This is quite a powerful program to use but you might need to read the manual to understand it. You can input the pixel size so that your measuring functions will output the data in the scale you want, for example µm. This software is freeware.

ImageTool http://ddsdx.uthscsa.edu/dig/itdesc.html
This is quite a powerful program to use but you might need to read the manual to understand it. You can input the pixel size so that your measuring functions will output the data in the scale you want, for example µm. This software is freeware.

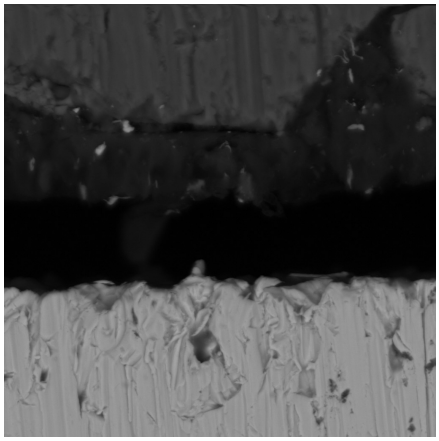
MeasureIT http://www.olympus-sis.com/en/6900_7026.htm (or ask the SEM operator if you've problems getting it)
This is quite a simple program that can put rulers and comments in a SEM image and then save it out. The pixel size is read directly from the SEM image so no manual input is necessary, as long as you're using a copy of the *original* image. Remember to choose 'Burn overlay into image' from Options when saving to retain all the measurements and notes. NOTE! I've noticed that when you save your images from the program, black will turn green if the image isn't true colour (24 bit). Ask the SEM operator to convert the images if you're having problems with the colours. This software is freeware.

Photoshop Ask your computer support for this.
If you're doing a lot of image editing, this program is a must!

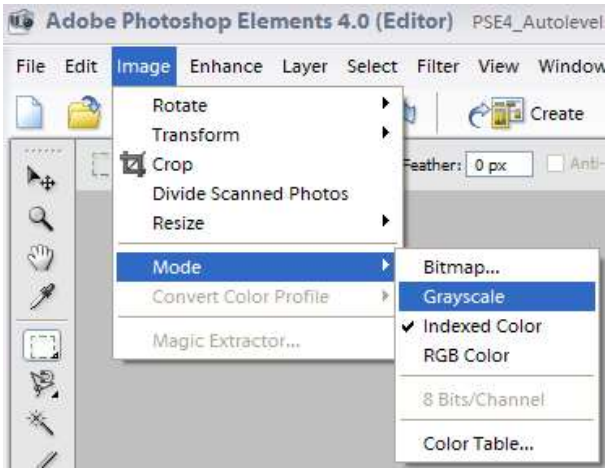
How to improve SEM images in Photoshop Elements

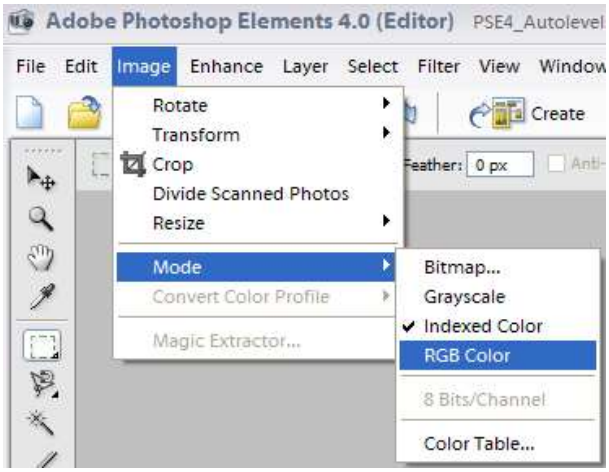
Sometimes it might not be possible to get the dynamic range with the SEM that you would like. You might wist that the images had a bit more contrast or were a bit sharper. Here is a small guide to help you improve on the quality of your SEM images using Photoshop Elements.

1. Load the original image into Photoshop Elements.

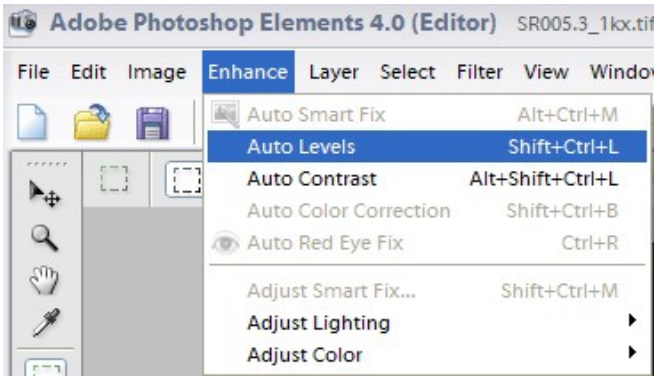


2. Choose “Grayscale” or “RGB Color” from the “Image->Mode” menu to convert the image.

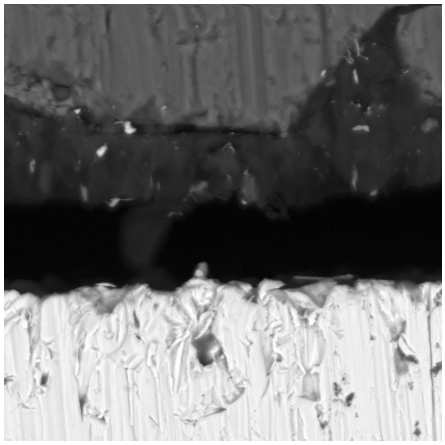




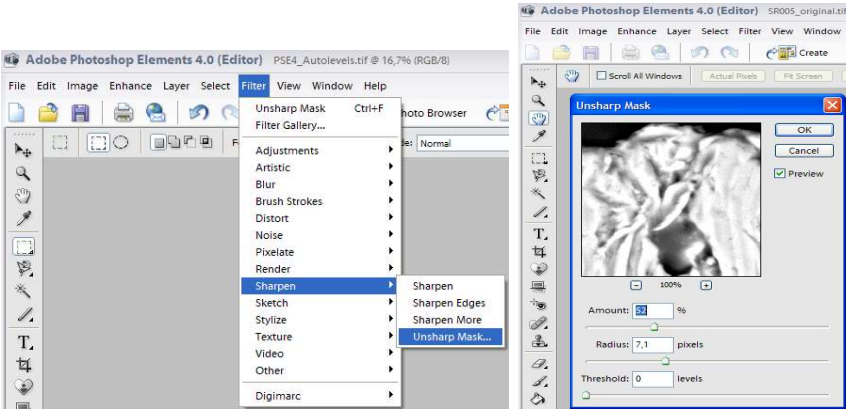
3. Choose “Auto Levels” from the “Enhance” menu. You can also try “Auto Contrast”.



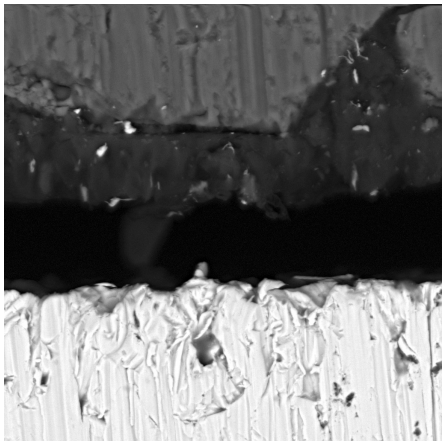
4. Your image should now look brighter.



5. Choose “Unsharp Mask…” from the “Filter->Sharpen” menu and play with the settings in the window that opens until you’re satisfied.



6. Your image should now look sharper. Save it!



Johan Gadolin
Process Chemistry Centre

ÅBO AKADEMI
UNIVERSITY

SEM work order

Name _____
Institution/Company _____
Address _____
_____

Telephone _____
E-mail _____

Project name _____
Project number _____
Requested date _____
Deadline _____

Comments _____
_____
_____

Sample name	What is it	What to do	Comments

Contact information

Mailing address:
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Finland

Please check out the next page for a pictorial on how to get to the SEM laboratory at Åbo Akademi University. Please don't knock on the door and wait for me to open it as I might not hear you, come straight into the laboratory. If the door is locked, please knock as hard as you can as I might have forgotten to unlock it.

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Work phone	+358 (0)2 215 3513

The work phone might not always work due to bad reception in the SEM laboratory, please try the SEM laboratory number first when calling.

Johan Gadolin
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