University of Ulm
Institute for Micro- and Nanomaterials

Lab
„Materials Science“

Summer Term 2007

Group 9: Adelheid Grob

F2: Microstructure II
lab on July, 19th 2007

1 Introduction

In this lab we took a closer look at material analysis done by SEM (Scanning Electron Microscope). We examined different probes with different analytic methods (SE, BSE and EDX). SE means the analysis with Secondary Electrons only; for this analysis we had two detectors, one is fixed in the lens, the other one at the sidewards to the probe. The sideward detector gives us a better topological view, whereas the in-lens-detector is much more efficent and gives as a sharper view on the probe. Both detectors are scintillization detectors, the in-Lens detector is working with an electrical lens, which collects the electrons coming from the sample, the sideward detector works with a collecting bias, which attracts the electrons.

The BSE (Back Scattered Electrons) analysis mode gives as more information about the consistence of the probe, but the information about topology is not very good, this is due to the fact that the energy range of BSE is very big. So often both modes, the SE and the BSE mode are used to get information of a sample.

In higher energy ranges even Auger electrons and/or x-rays appear, the characteric x-ray lines can be used to get information about the composition of the material. This analysis is called EDX-analysis.

In our experiment we performed all described ways of getting information of the different probes.

2 Au-C-Sample

The first sample we took a look at consisted of Au-C-Clusters. This probe is a standard probe, which is often used for checking the SEM.

As the energy of the secondary electrons is directly proportional to the atomicity Z of an element, we would expect gold to be quite bright (Z=79) and the carbon to look quite dark (Z=6).

First we took a look at the whole probe. We saw the C-lattice (looks black) and some black lines at the surface, which are due to the fact, that the probe is affected by outer effects (see fig. 1).

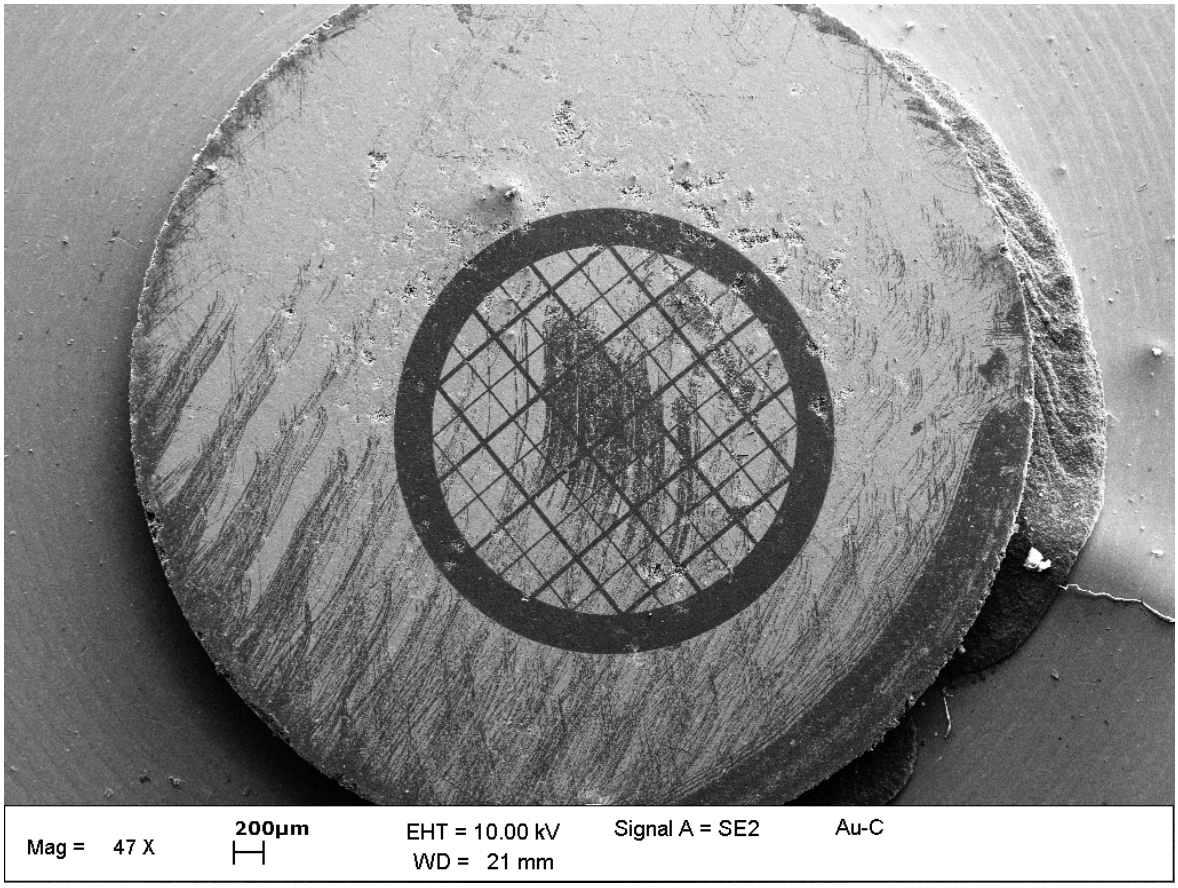

Fig. 1: Whole Au-C-sample

The next thing we did is to zoom into the probe by decreasing the working distance to 5 mm. We went on decreasing the working distance until we've got a focussed magnification of about 500. At this magnification we could see the gold clusters (the white particles) and the carbon (dark particles). You can see the result we got from the In-Lens-Detector in Fig. 2.

Afterwards we tried to find out the difference of the picture we get with the In-Lens-Detector and with the sideward-mounted detector. As you can see in Fig. 3 the result from the In-Lens-Detector (the left one) is much clearer than the result we got from the sidewards detector. The Signal-to-noise-ratio (SNR) is about dimension better if the In-Lens-detector is used.

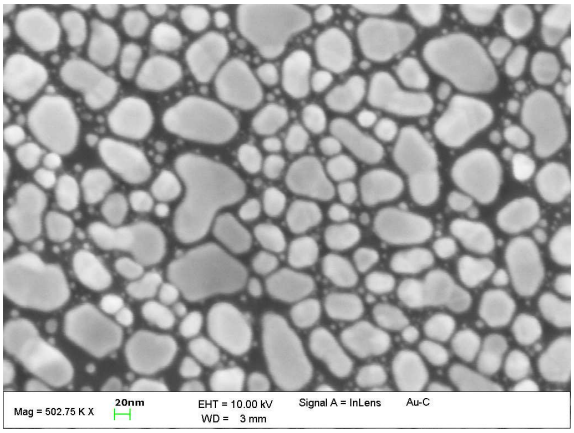

Fig. 2: Au-C-sample, In-Lens-Detector: Gold Cluster on carbon

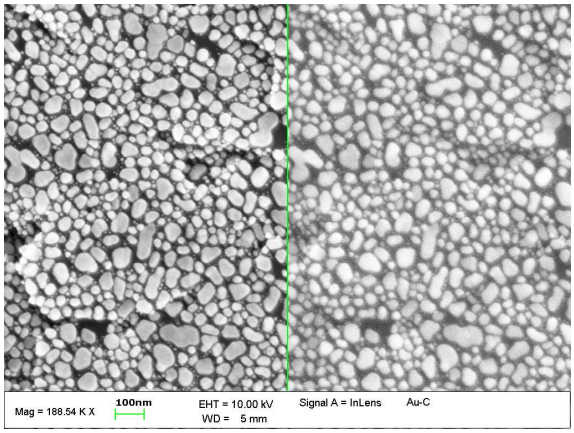

Fig. 3: Au-C-sample: Comparison In-Lens-detector and sideward detector

3 Low-Carbon-Steel-Sample

In the next part of the experiment we took a look at the Fe-C-probes we prepared in lab F1 (Microstructure I). First we examined the low-carbon-steel-probe.

The first picture (Fig. 4) we took was with the sideward detector at a magnification of 2000. It showed us an overview of the structure. We can see the grain boundaries (the small lines) and regions of different phases - pearlite and ferrite. Then we changed to the In-Lens-detector and zoomed in to a pearlite lamella: see Fig. 5. The picture was taken at a magnification of 20000 with the In-Lens-Detector.

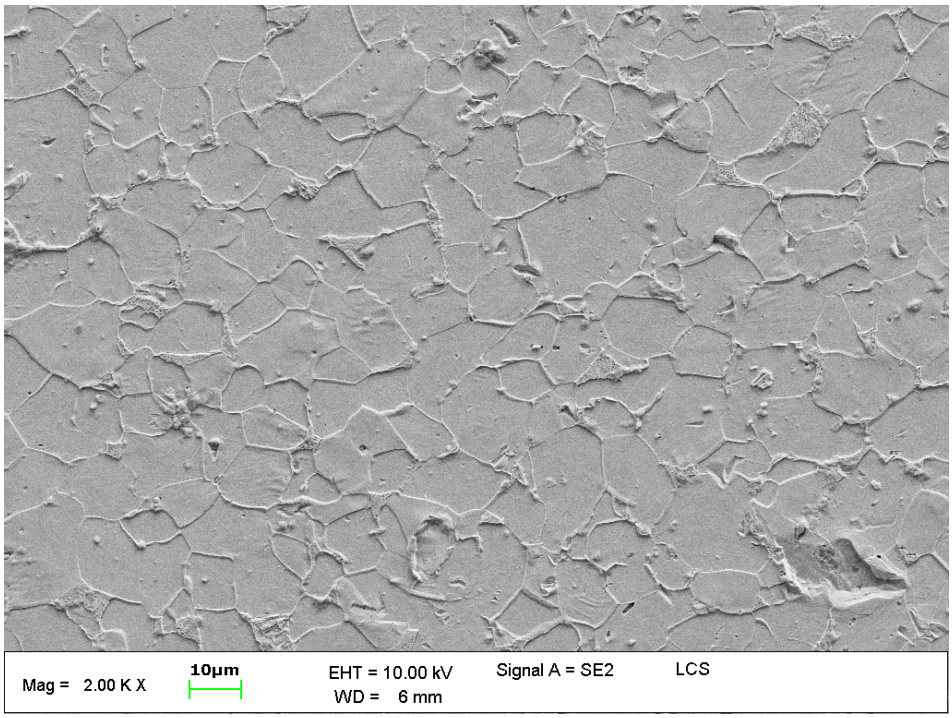

Fig. 4: Low Carbon Steel: sideward detector

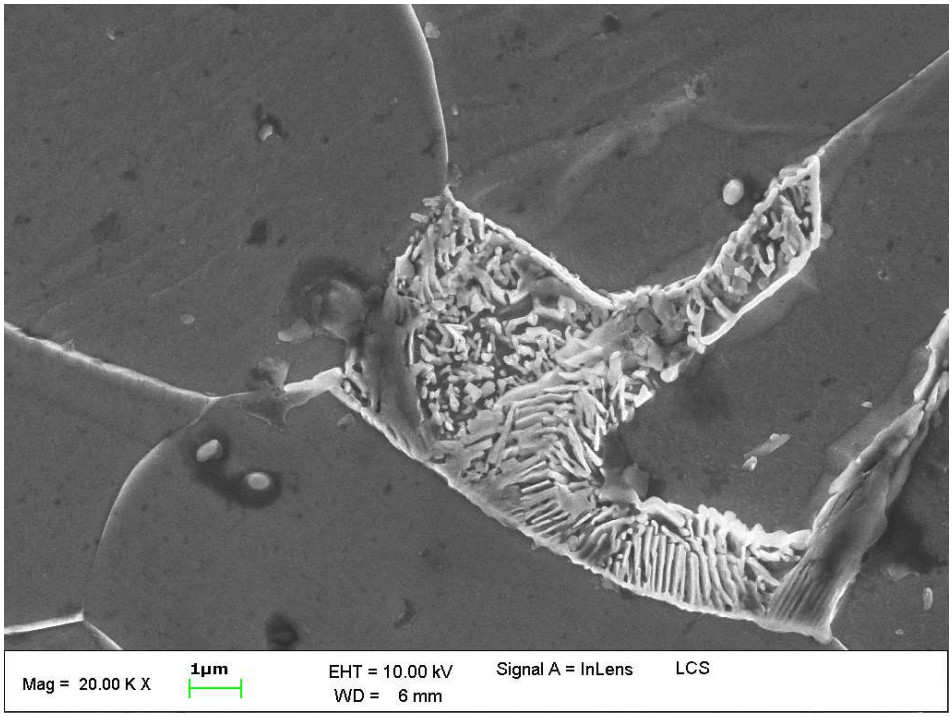

Fig. 5: Low Carbon Steel: pearlite lamella

By taking a picture at a magnification of 1740 with the In-Lens-Detector, we can see, that the contrast in this picture (see Fig. 6) is much better compared to the Sideward-Detector-Picture, but we aren't able to get any topographic information about the structure with the In-Lens-Detector. So what we did to get is to combine both signals in order to get a picture with a good contrast and a good topographic information. The result you can see in Fig. 7.

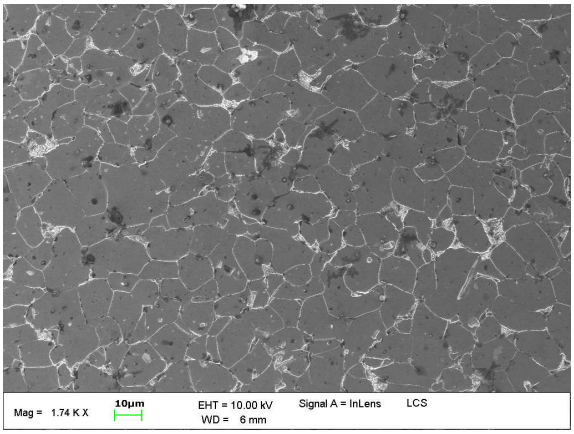

Fig. 6: Low Carbon Steel: In-Lens-Signal

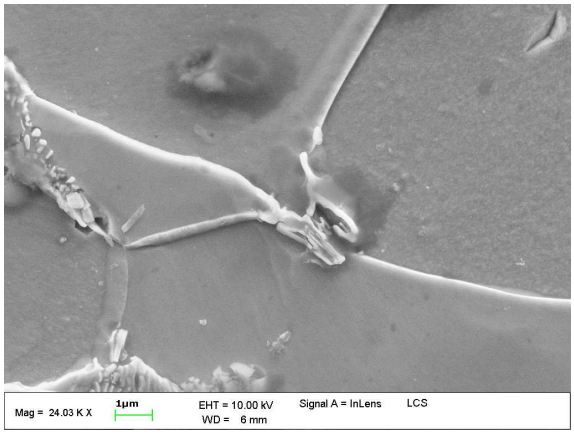

Fig. 7: Low Carbon Steel: Combined Signal

4 High-Carbon-Steel-Sample

The next sample we looked at was the High-Carbon-Steel-Sample we also prepared in Lab F1. First we took again one picture with the In-Lens-detector and one with the Sidewards-Detector (Fig. 8 and Fig. 9).

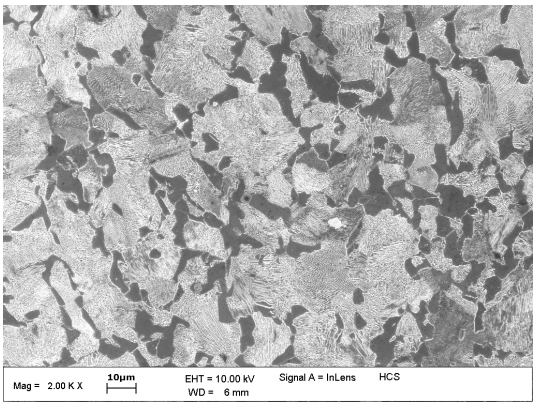

Fig. 8: High Carbon Steel: In-Lens-Signal

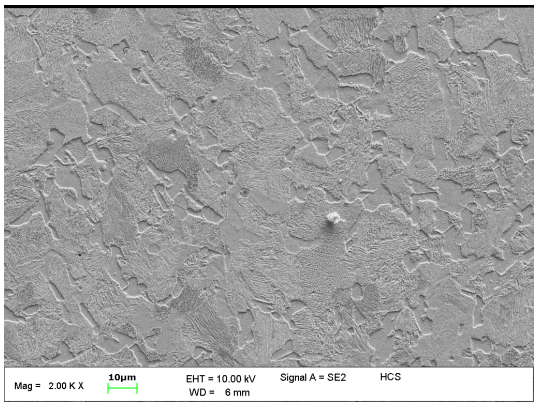

Fig. 9: High Carbon Steel: Sidewards-Signal

Now we zoomed in to a magnification of 15000 K and took a picture with both signals. The result can be seen in Fig. 10. What we can see in this picture is a defect in the FeC (the squared hole) as well as the pearlite lamellas again and the ferrite phase. As we expected and as we have seen already in Lab F1, there is more pearlite in the sample due to the effect that the sample contained more carbon.

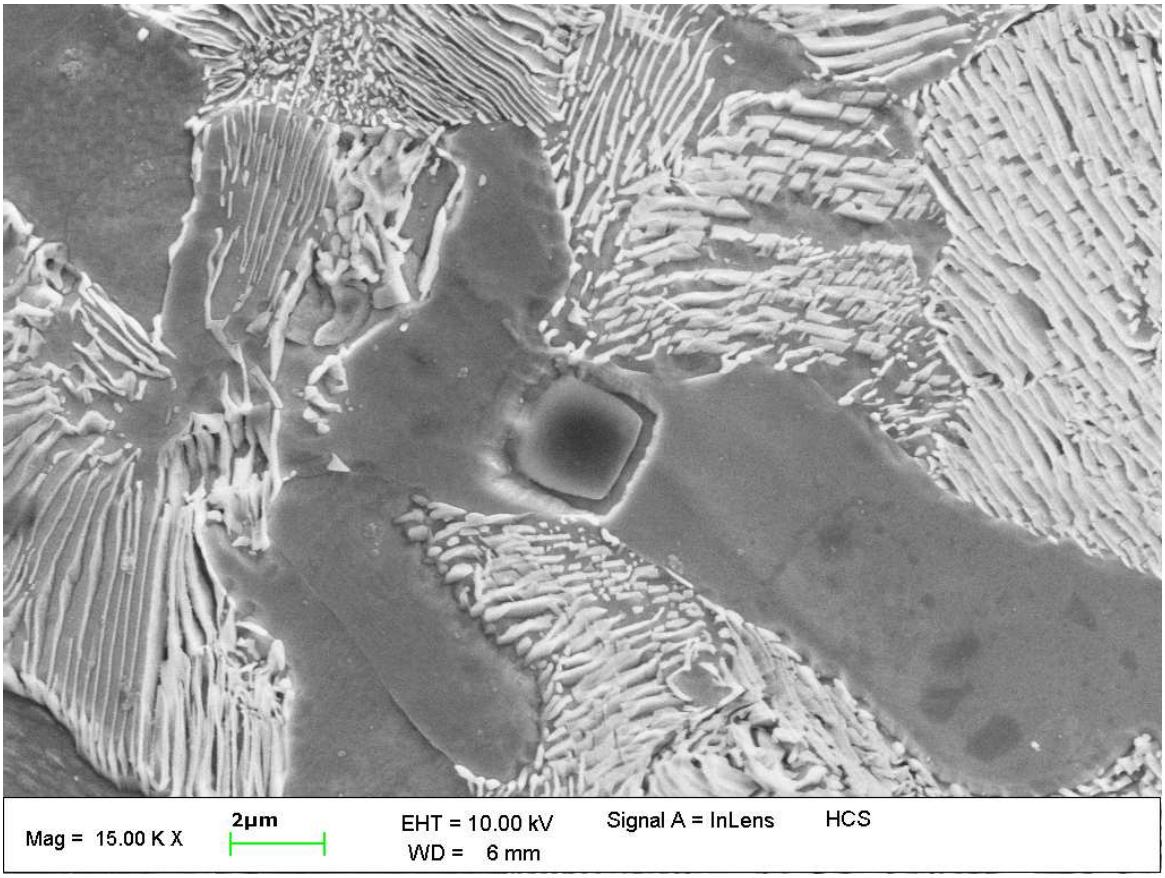

Fig. 10: High-Carbon-Steel: Both signals

At the end we went to a magnification of 84000 to see the pearlite structure more clearly. We can see now the single stubs of the lamella (see Fig. 11).

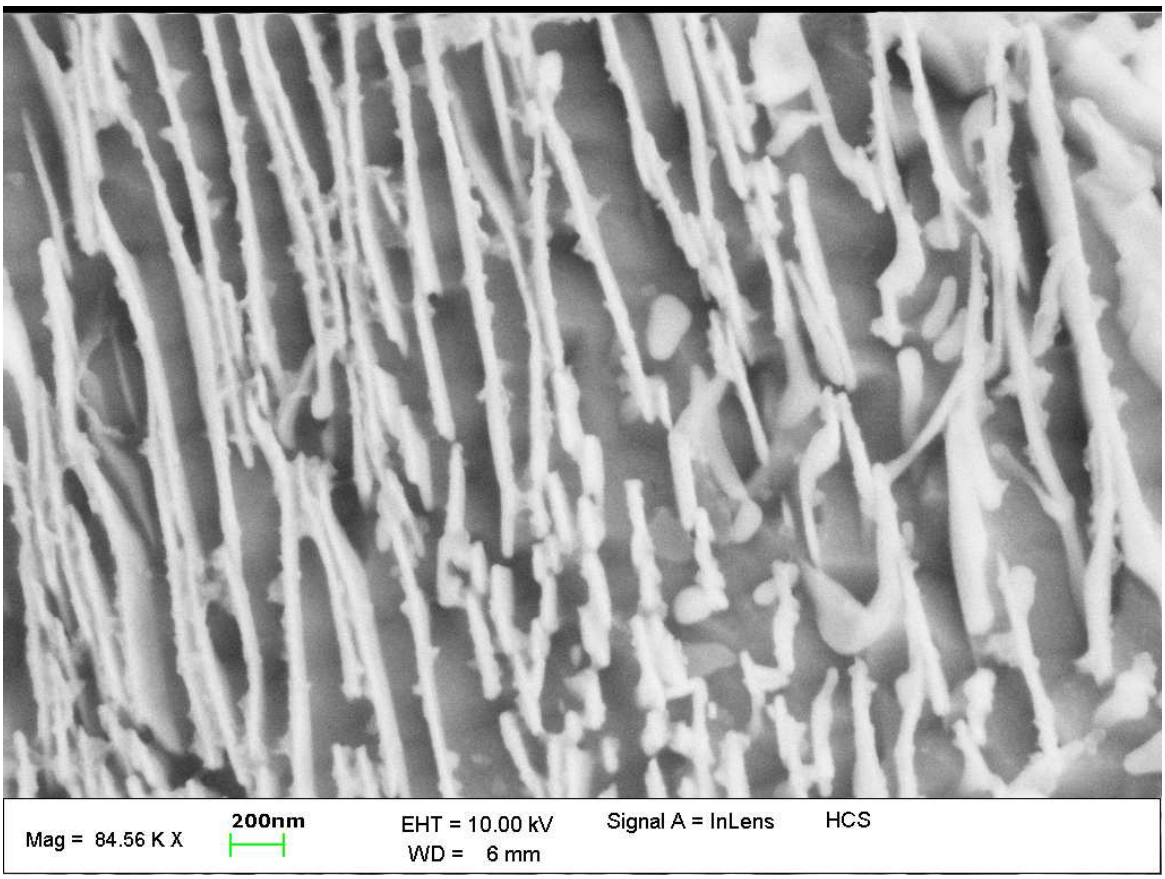

Fig. 11: High-Carbon-Steel: Lamella Structure

5 Sn-Pb-Cu-Sample

In the last part of this lab we examined a sample which contained of leadtin with 2 weight% copper. With BSCE- and EDX-analysis we tried to find out the percentages of lead (Pb), tin (Sn) and copper (Cu) in our sample. Therefore we searched first for a section which we held quite typical for the probe, because the EDX-analysis can only done for a certain section.

The section we took for the further examination is displayed in Fig. 12.

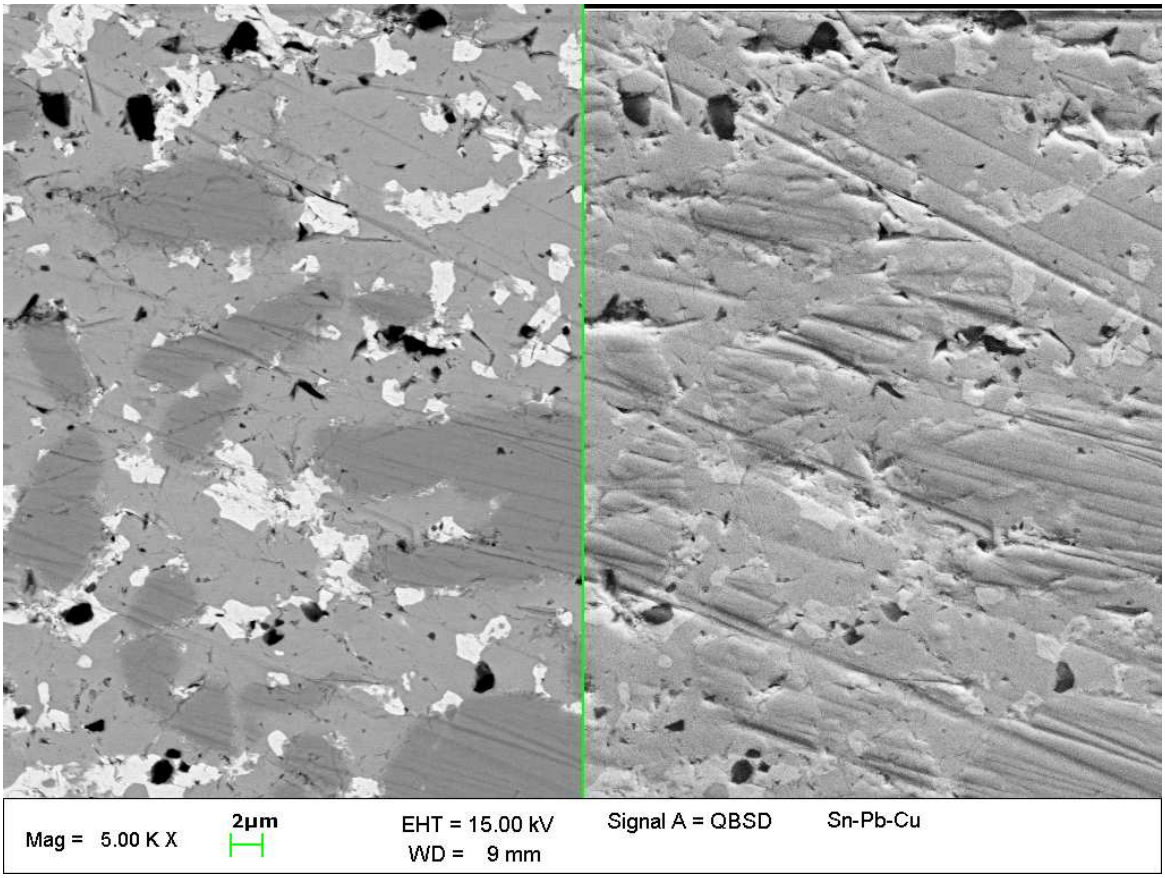

Fig. 12: Sn-Pb-Cu-Sample: selected section

In the right half of the picture you can see the signal from the SE and on the left the signal from the BSE. It can be seen, that there are three different regions: the light one, the dark and light gray one. Now we use the X-ray signals which are detected to find out which region is what.

This can be done, because every material has different spectral X-ray-lines, which are well-known. So we mark each region we want to examine (you can see the small numbers in Fig. 13). So the programm delivers us the following values for each region:

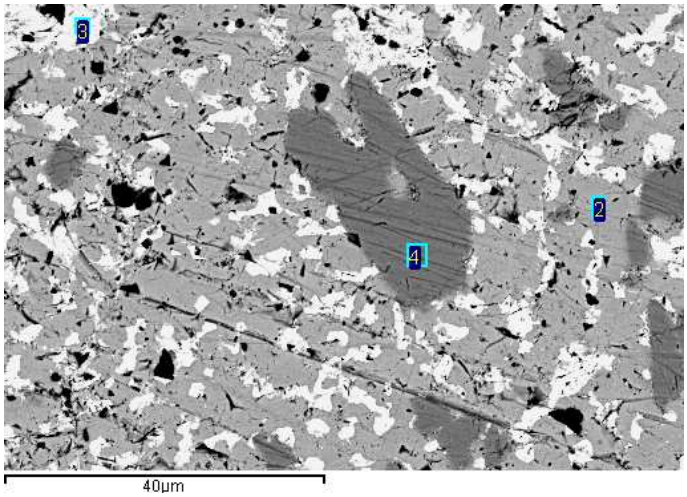

Fig. 13: Marked regions in the probe section

region	wt% Cu	wt% Sn	wt% Pb
2	0.44	98.87	24.99
3	-0.16	4.71	95.45
4	37.01	63.05	-0.05
total	2.19	72.82	24.99

Table 14: weight percentages of Cu, Sn and Pb as measured from Fig. 13

As we can see in table 14, region 2 (the light one) contains mostly of tin, region 3 (the light gray one) is nearly pure lead, whereas region 4 (appears in dark gray in Fig. 13) contains out of tin and copper in a ratio which nearly corresponds to 3:2. This result is what we expected as copper and tin form in Cu_3Sn_2-constellation.

In total the result gives as a value of 2.19 wt% copper, what is nearly the value we assumed in the beginning. That means the section we've chosen was a good one.

The ratio of lead and tin is 1:3.

We also found a slight amount of Silicium in our probe, which comes from the preparation of the probe, it was grinded with a SiC-Paper.

Next thing we did was to scan over the section again (the examined section is drifted a little bit in comparison to the first examined section, as you can see) and we tried to find out which phases are present.

The scanning programm allows us to show the different phases in different colours, as you can see in Fig. 15. If we plot the percentual distribution of the phases we get the diagram shown in Fig. 16.

We can see, that the sample consists of 10.31 % of phase 1, of 66.55% of phase 2 and of 19.98% of phase 3. That means that the tinrich phase (Phase 2) is the most present in the sample, while the mixed tin-copper-rich phase has the least amount in the sample.

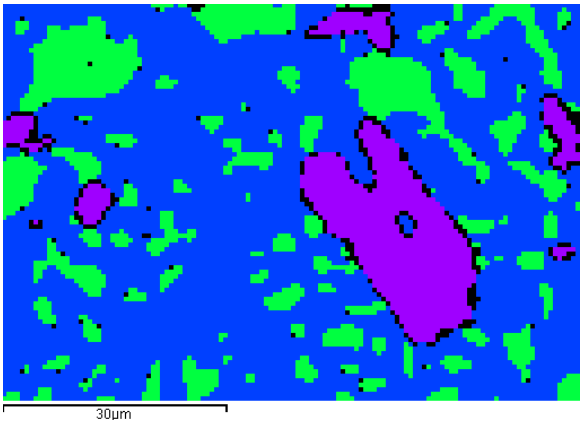

Fig. 15: Different phases in sample section

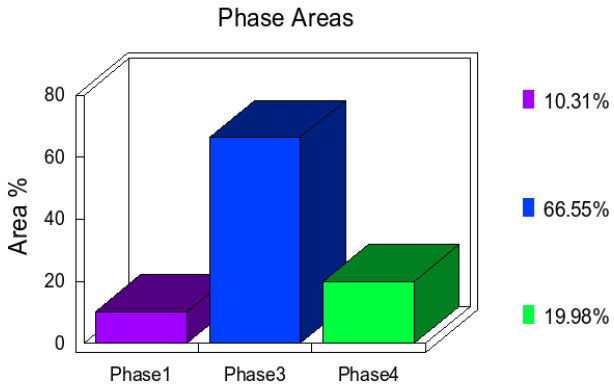

Fig. 16: Percentual distribution of phases

The last thing we did was a again a mapping over the sample section and to plot now different pictures for the different x-ray-lines of the three components Cu, Sn and Pb. The result is shown in the Fig. 17 to 20. This method also shows us where which material is concentrated.

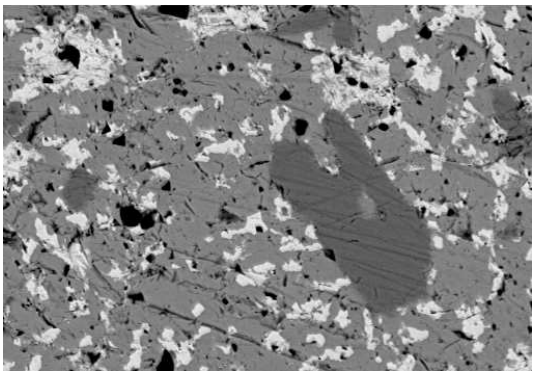

Fig. 17: The whole sample section

Fig. 18: The copper Kα1-line

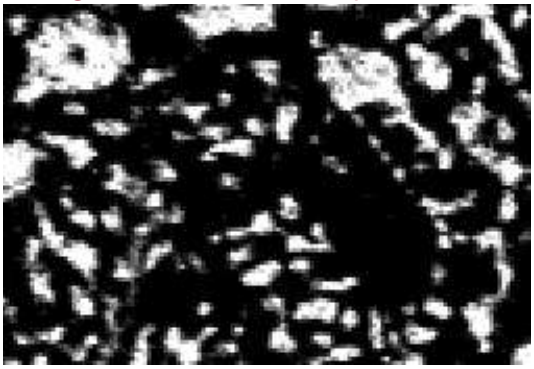

Fig. 19: The lead Mα1-line

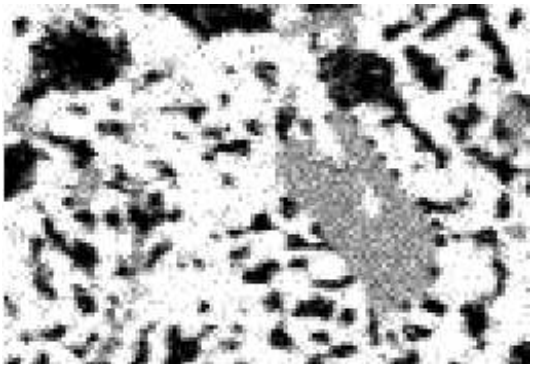

Fig. 20: The tin Lα1-line