DESY Summer school 2007

Kirsi Leppänen

University of Helsinki
kirsi.leppanen@helsinki.fi

Supervisors Thomas Wroblewski and Andre Rothkirch
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1 Introduction

A new X-ray imaging method was tested and studied, where between the sample and detector 300 mm long capillary bundles were used. Experiments were meant to be made by using white beam which includes all X-ray wavelengths. The aim of using the capillaries was to find a new way to suppress scattering caused by polycristalline samples.

Measurements were conducted using an old spear head made of iron as a sample. The head of the spear was in three pieces: the pieces were different in their sizes and thicknesses and the third part was the very top of the spear. All of them were very rusty and covered with layers of corrosion and soil with traces of plants. So by using only eyes it was impossible to see any signs of the structure or the surface of the spear head.

1.1 X-Radiography

X-radiography is an investigation technique which is fast and non-destructive and this latter thing is extremely important when considering the investigation of archaeological samples. It reveals the structure, form, size, surface features and details of the sample which can be heavily or completely corrosed or under a thick layer of burial accrecations and organic substances. Radiography does not cause any mechanical or other physical or chemical effects to the object and for some type of samples X-radiography can provide information that could not be gained by using any other method. A good quality radiography result provides the information which is needed to identify, classify and date the sample [6]. In radiography measurement the sample is exposed by X-rays and the image is generated by the transmitted X-rays, so the image comes about the variations in absorption due to thickness and composition of the sample. Scattering from the sample causes problem, because it blurs out the image.

1.2 Polycapillaries

Polycapillaries offer the possibility to increase the sample to detector distance without the loss in resolution. Polycapillaries are manufactured by pulling heated glass. The starting point of the polycapillary structure is a single glass tube, which is pulled to a very thin capillary. These capillaries are collected into bundles and pulled repeatedly into the wanted final form. The relative large cross section, which is needed for measurement purposes, is gained by collecting polycapillaries into larger bundles and fusing them by thermal treatment into a monolithic structure [1, 2]. The super structure of capillary bundles in the form of the hexagonal arrays can be well detected in some of the measurement results. The aspect ratio of the used capillaries was 10^{-4} in this measurement.

1.3 Samples

The sample was an old spear head from Poland. The sample was kept in three pieces, and those pieces can be seen in the figure 1. In the figure 1 there can be seen all the three parts in that order as they are meant to be together: the smaller part, the bigger part and the very top of the spear head. As can be seen from the photo the pieces were covered with soil and rust. The pieces were found in the ruins of medieval castle in Czersk near Warsaw. They were found in the grave which belongs probably to the Masovian Prince, and they are dated for the end of the 12th century. In the same grave were found also other objects [8]. The archaeologis were afraid of removing the layers of dust away from the samples, because the samples are so old and easily damaged. So there was a real need for the X-ray investigation of these samples.

Figure 1: The measured sample as a whole.

2 Theory

2.1 Diffraction Enhanced Imaging: Rocking curve

In Diffraction Enhanced Imaging the used beam is monochromatic and the beam goes to detector through an analyser crystal. Bragg's condition is fullfilled only when the incident beam is in correct angle with the lattice planes of the sample for a certain X-ray energy. In this case the beam diffracts over a narrow range of incident angles. With analyser crystal it happens, that the diffracted intensity will follow the path of rocking curve [3, 4] when the analyser crystal is rotated. We wanted to see if this phenomenom would be detectable in our experiment which was done by white beam and using the capillaries, so the capillaries were slightly turned during the experiment. When the capillaries are rotated about a horizontal plane, the same should happen, which can be observed normally with analyser crystal.

2.2 Suppression of scattering

In polycristalline samples there happens a lot of scattering, which harms the imaging, because it blurs out the image. That is why it is wanted to suppress the scattering and in this experiment it was tested by using the polycapillaries between the sample and detector.

2.3 Handling images by IDL

Scanned images from image plates were handled by IDL. I wrote short IDL programs which took care of the basic image procedures like calculating the signal to noise ratios or center of masses of the images, mapping the images, subtracting the backgrounds and comparing the statistics of the images. By these simple programs the image plate results could then be analysed. The results of the measurements with smaller part of the spear head are in the appendix A.

The problem in the analysis was overexposure of the image plate: the images suffered from the high sensitivity of the image plate, so that many of the pixels were in the saturation value and it was impossible to get any information out of that kind of pictures. Also mapping was a difficult task, because scanned images were always in slightly different positions and taken by different conditions so that the intensity values changed drastically from image to image. So there was no simple certain type of solution to the mapping problem, which would work in all cases.

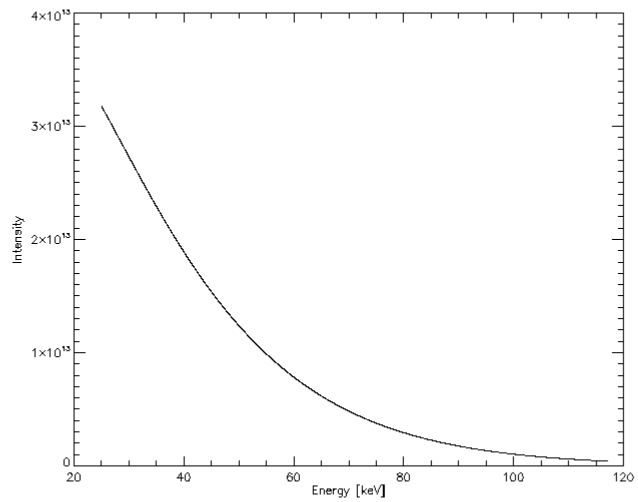

Figure 2: The spectrum of the beam without tantalum.

2.4 Calculations of the X-ray spectrum

The spectrum of the used synchrotron beam had to be calculated. For the calculation was used already existent program, which can be found from the HASYLAB web pages [5]. The values for the absorption coefficients for the tantalum were taken from NIST web page [7].

The calculated spectrum from the program is the spectrum of bending magnet and it gives the distribution of radiation emitted by electron. The energy of the electrons was 4.44 GeV, the storage ring current was $90 - 140$ mA, the magnetic field of the benging magnets was 1.2182 T and polarization was S-polarization, which means, that the electric field is in the plane of the ring. The absorption coefficient values had to be multiplied by the mass density of tantalum ρ. The intensity follows the law

$$I = I_0 \exp\left(-\mu \rho d\right), \tag{1}$$

where the thickness of tantalum is marked by d. As a result a spectrum of intensity values against energy values was gained and the results can be seen in the figures 3, 4 and 5 for the tantalum thickness 0.1, 0.4 and 1.0 mm respectivily. The original spectrum can be seen in the figure 2. There can be seen a dramatic difference between these spectra. Tantalum has its K-edge around 67 keV, which causes the shape of the spectra.

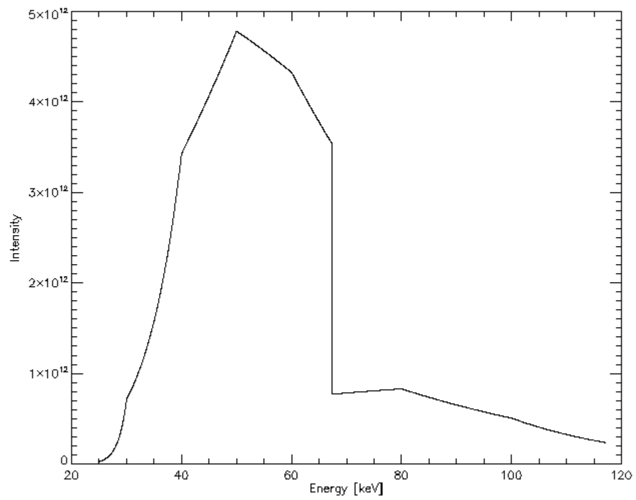

Figure 3: The spectrum of the beam with tantalum thickness 0.1 mm.

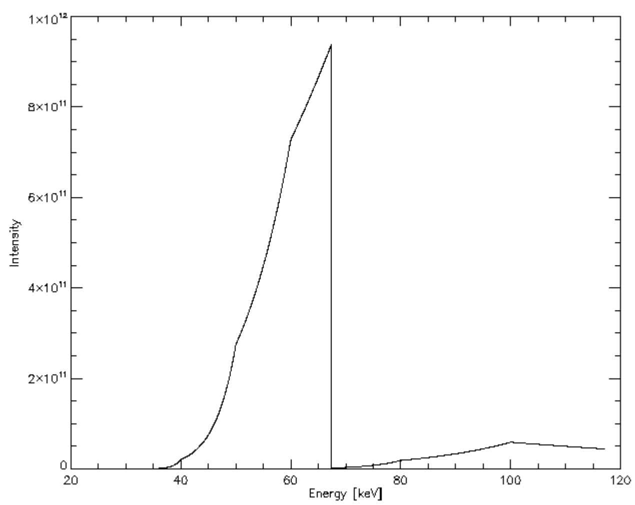

Figure 4: The spectrum of the beam with tantalum thickness 0.4 mm.

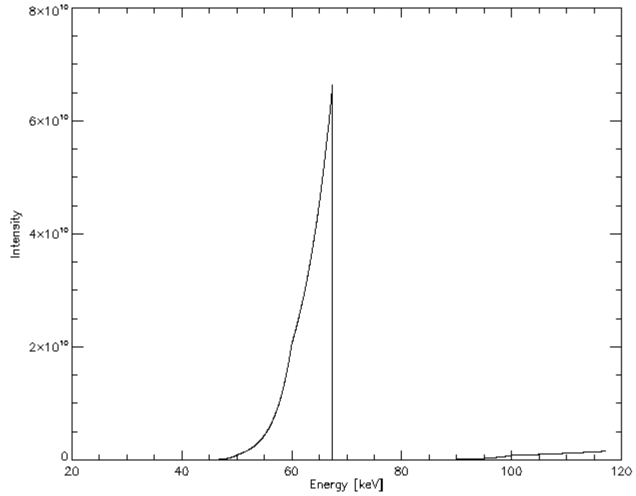

Figure 5: The spectrum of the beam with tantalum thickness 1.0 mm.

3 Measurements

The first actual measurements I made in HASYLAB were conducted using ordinary X-ray tube and image plate. These measurements I made just for the case of learning how to handle image plate: how to scan it and to see how it behaves as a function of the radiation. Aerogel was used as a sample and at that time it was planned that it would also be used as in the actual measurements. That never realized because the sample is strong scatterer, and it would have been impossible to measure it by our methods where hard beam was used.

The synchrotron radiation measurements were conducted using the set up at the HASYLAB beamline F3. The set-up consisted of slit system, sample holder, polycapillaries, image plate/film holder and image plate or film was used as a detector. Schematic picture of the used set-up can be seen in the figure 6. All the parts of the set-up like sample and film holder, slits, capillaries and collimator were moved by step motors and these step motors were handled through the program called Online. The cables connecting step motors and track had to be checked and ordered before the measurements started. For that purpose we wrote a simple table, which tells the motor numbers, cable number and the alias in the Online program.

The used beam size was as large as possible, which meant an area of 6 mm times 6 mm. That caused immeadite problem with the safety level of the radiation and that had to be solved by using many lead shieldings and coverings around the whole system. Extra safety measurements were

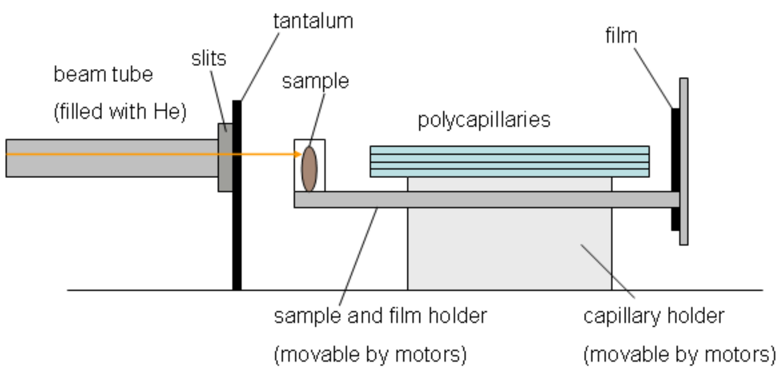

Figure 6: The schematic picture of the used set-up.

conducted around the hutch, and they showed that the radiation there was in the same level as the level of the normal background.

The measurements started with carefully conducted alignment measurements. The capillaries had to be well aligned with the beam, and for that reason a two dimensional scan was made in the plane of the capillaries and 2D-gaussian was fitted to the this data. Capillaries were then moved to the position of the intensity maximum.

3.1 Measurements with image plate

The sample holders were made out of plastic, and they had two different sizes; the smaller and bigger pieces were kept in another sample holder and the top of the spear was kept in thinner sample holder. Sample holder was amounted to the same device as image plate holder so that they could be moved simultaneously and the whole area of interest of the sample was easy to scan. The scanned areas were about 100 x 40 mm^2 for the biggest part and 50 x 40 mm^2 for the smaller part. The scanning steps in horizontal and in vertical directions were changed between 1/2 and 1/3 of the beam size so that the optimal exposure could be found. If the scanning step size was 1/3 of the beam size, it means that every point was exposed three times. An automatic beam shutter and preset count mode were used in every image plate experiment. The longest scanning took over two hours in the case of the biggest piece, but the average measurement time was one hour. Measurements were made both with the capillaries and without the capillaries. The alignment of the capillaries was also slightly shifted between the measurements which were made with capillaries. Some of the results of comparisions in the case of the small piece of the sample can be found in the appendix A.

There were lots of problems with image plate and the intensity of the white beam, because many of the pixels of the image plate were in the saturation point after short exposure to the beam. Some of the problems were solved by using lead plates in front of the beam, but this meant at the same time, that the beam was not any more white beam, because lead cut off the lower parts of the spectrum.

3.2 Measurements with films

To avoid the using of thick lead plates in front of the beam the image plates were substituted by ordinary films. Film has the advantage that it is less sensitive compared to the image plate, which easily ends up with saturation level. But also the films could not be used totally without anything in front of the beam: we used tantalum-plates with thickness of 1.0, 0.4 or 0.2

Property	Kodak film	Slavich film	Fuji image plate
Size	13x18 cm	10.2x12.7 cm	20x25 cm
Resolution	50 μm	3000 lines/mm	50or100or200 μm
Sensitivity	$<$ Slavich	at 530nm:75mJ/cm^2	« Slavich
Max. of spec. sensit.	?	530 nm	?
Diffraction efficiency	?	at 530 nm: $>$ 40 %	?

Table 1: The properties of the used films and image plate.

mm. Measurements with films were made without beam shutter, so that the scanning was going on continuously. The used vertical scanning step size determined the amount of the beam exposure. Many step sizes were tested before the optimal size was found for every different piece of the sample. Measurements were made both with the capillaries and without the capillaries. All the important parameters of succeeded measurements can be seen in table 2.

Two kinds of films were used and the properties of the two films can be seen in the table 1. Also some of the properties of image plate are compared with films. Kodak film (Kodak Industrex SR45) had higher sensitivity but lower resolution compared to the another film (Slavich VRP-M), but the first really good results were gained by using Kodak films. After determining the right exposure time good results were got also by using the other film type. Films were kept in the developer mixture for 4 minutes, after it they were shortly dipped into diluted acetic acid and from there to fixing mixture. After about another 4 minutes in the fixing tank, they were washed with water and then dried. First three steps were absolutely important to be made in dark room.

4 Results

Results of the measurements can be seen in the figures 8, 9, 10, 11, 12 and 13. The most important measurement parameters are mentioned in the table 2.

5 Conclusions

First experiments using image plates failed and showed severe problems, because they were too sensitive for the intensity of the beam and the resolution of the plates was not as good as might been hoped. Because the saturation limit was achieved so easily, we could use only very rough stepping and that caused the stripe pattern to the many of image plate results (see for example figure 7). So there were many

Figure 7: The picture of the small piece taken by image plate. The picture can be compared with the image taken by X-ray film which can be seen in the figure 10. The resolution and the sensitivity of the image plate are not as good as those same properties of X-ray film. This image is taken without capillaries.

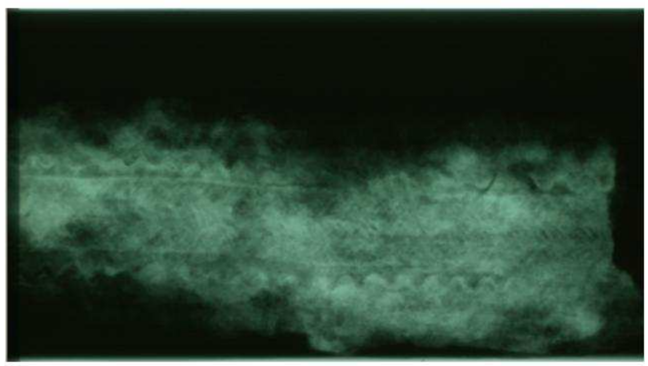

Figure 8: The first succeeded exposure. Image is taken from the biggest part of the sample. There was 1.0 mm tantalum in front of the beam and polycapillaries were in use.

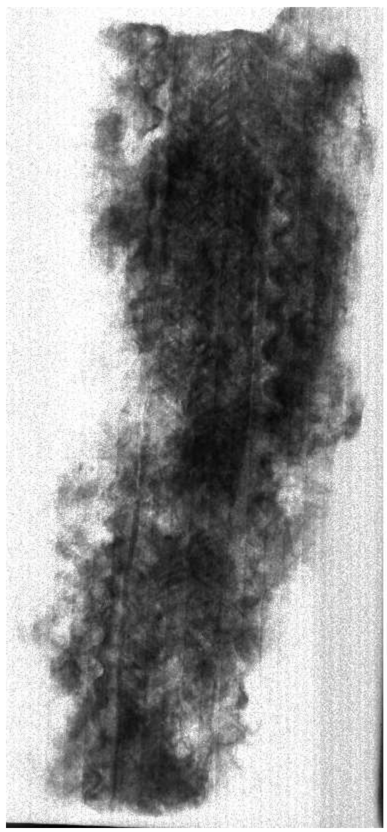

Figure 9: Image of the biggest part of the sample taken without the capillaries.

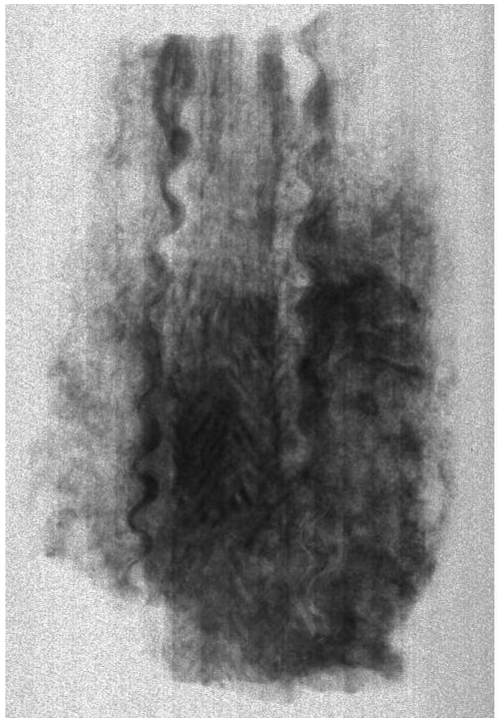

Figure 10: Image of the smaller part of the sample taken without the capillaries.

Figure 11: The top of the spear head taken without the capillaries.

Figure 12: The detail from the former picture 11.

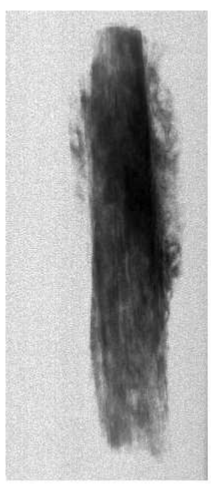

Figure 13: The image taken of the side of the smaller part of the sample.

image	film type	thickness of Ta [mm]	vertical step [mm]	note
8	Kodak	1.0	1.0	with capillaries
9	Kodak	1.0	2.0	without capillaries
				beam size:0.5x6 mm
10	Kodak	1.0	2.0	without capillaries
				beam size:0.2x6 mm
11	Slavich	1.0	0.1	without capillaries
13	Kodak	0.4	1.0	without capillaries
				beam size:1x6 mm

Table 2: Table of the results and information included in them. If the size of the beam is not mentioned it is 6 x 6 mm.

reasons to use films instead of image plates and good results were achieved by using films as detectors.

Capillaries between the sample and film did not bring any improvements to the results. The reason for that was the fact that there was tantalum in front of the beam, so there was no white radiation in reality. Scattering is caused mainly due to the low energy part of the beam spectrum, and now this part was cut off and only the high energy part of the spectrum did pass the sample. The capillaries had the effect that they damped the intensity of the transmitted beam on the film, but that was the only thing what they did. There was no such amount of scattering that the imaging would have been impossible without capillaries. So in the end the images were taken with thin tantalum plate ($0.4 - 1.0$ mm) in front of the beam and without capillaries and the results were good.

In each of the images a clear pattern can be seen in every piece of the spear head, so the pattern continues through the whole sample.

Appendix A

IDL-results with image plate measurement of the small piece

Mapping was done by calculating the center of masses of the images and then choosing the similar areas around the center of masses and in this case this worked out well. Three different images were compared with each other: image taken with capillaries, image taken without capillaries and image taken with capillaries which are a little bit shifted from the aligned position.

The results of statistical calculations:

image	mean	variance	median	minimum	maximum
with capillaries	26202.5	3478.95	26106.0	14967.0	35707.0
without capillaries	18845.1	7168.55	20212.0	3477.0	35813.0
shifted capillaries	8744.35	5862.84	5436.0	1688.0	29052.0

Table 3: Table of the results calculated from image plate measurements.

References

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[4] Li J., Zhong Z., Lidtke R., Kuettner K.E., Peterfy C., Aliyeva E., Muehleman C., J. of Anat. 202(5) (2003) p. 463-470

[5] http://hasylab.desy.de/facilities/sr_and_fel_basics/sr_basics/index_eng.html

[6] http://www.english-heritage.org.uk/upload/pdf/X_Radiography.pdf

[7] http://physics.nist.gov/PhysRefData/XrayMassCoef/tab3.html

[8] Wladyslaw Weker's private communication