ESRF	**Experiment title:** Three dimensional structure of paper studied by microtomography	**Experiment number**: ME-55
Beamline: ID22	**Date of experiment**: from: 10.05.00 to: 14.05.00	**Date of report**: 22.02.01
Shifts: 12	**Local contact(s)**: Timm Weitkamp	*Received at ESRF:*

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Report:

Background

The three-dimensional structure of paper is important for many paper properties like absorption, optical properties, permeability, strength and compressibility. A more accurate knowledge of the dependency between the paper components characteristics, paper structure and paper properties can help the paper manufacturers in optimising the structure for certain end use properties. Scanning electron microscopy of cross sections is a common method for structural investigation of paper structures [1]. Recently high-resolution x-ray microtomography has shown up is a very promising method for making 3D images of paper in microscale [2]. The high resolution (0.35 μm) and the automatic reconstruction make this into a feasible method for making 3D representations of real paper samples.

Experimental conditions

See beamline information in Appendix 1.
High resolution Frelon camera: 2048 x 2048 pixels

Performed work

Structure description

A series of different papers were chosen for the experiments, see Table 1. The first 11 samples were selected to cover a large range in papers structure configurations and tested in the state. Some of the 11 papers were singled out and soaked in water to be studied in wet state, see sample 12-15. In addition one filter paper were soaked in silicone oil before scanning to identify the possibillity to reach a higher absorption for the silicone filled spaces inside the paper structure.

Conductive polymers

The last 5 samples were performed in co-operation with Prof. Emil Samuelsen at Dept. of Physics (NTNU). His field of interest is electrical conductive polymers, which has to be oriented to be useful for

particular applications (e.g. LED). He was interested in studying how these solved polymers (polythiophene) were distributed in the capillaries. For this a filter paper structure were applied. The paper was soaked in polymer/toluene solutions with different polymer concentrations, see sample 17-21 in Table 1.

After the experimental run in May 2000, the method for reconstructing the images has been established at the Norwegian Pulp and Paper Research Institute. In the end of February 2001 all the reconstruction were finished, see Figure 1. Using the Interactive Data Language (IDL) and Dynamic Imager we have also converted one of the volumes into a binary format, see Figure 2. This was solved by choosing suitable thresholds and filters to determine the phase borders. A procedure to identify all voxels belonging to the same object (fibre) was written and all fibres were "filled" appropriately.

Planned and ongoing work

The results will be compared with data obtained from the paper 3D-structure images obtained by means of controlled grinding and SEM/CLSM.

Distribution of pore and fibre dimensions will be recorded from the data, and results will be used to calculate light scattering in paper by means of a light scattering model developed at ACREO, Sweden. In addition the data are used in a co-work with Prof. Silvy at UBI, Portugal, where the goal is to relate the mean cord length in the 3D porous paper structure to paper properties.

Further on the binary data structures will be used to build a model of the paper structure. This will be used for geometrically visualisation of the paper structure, but the final goal is to make a geometrical model with possibilities for directly modelling of light scattering/absorption and mechanical properties.

The important last step is to perform the analysis on papers made on a real paper machine with carefully chosen parameters to see how they influence the final paper structure. Such study will be a very important contribution to the knowledge about how to optimise the paper producing process regarding desired end use properties of paper. Beam time for this experimental series will be applied for before the next deadline for submission of applications (September 1, 2001).

Planned publications

The ongoing work together with Prof. Silvy is planned published in the immediate future, and as follow-up to ref.[2] we have planned to publish the procedure from a reconstructed image to the final binary image. The planned visualisation and modelling will also be included into this series of publications, and finally the planned study on relations between paper producing process and the final paper structure will be published.

References

1. Gregersen, Ø.Y., and Niskanen, K., *'Measurement and simulation of paper 3D-structure'*, COST E111, at PTS, Munich, December 1-3 1999

2. E.J.Samuelsen, P.J.Houen, Ø.W.Gregersen, T.Helle, C.Raven, *Three-dimensional imaging of paper by use of synchrotron x-ray microtomography*, Journal of Pulp and Paper Science, in press.

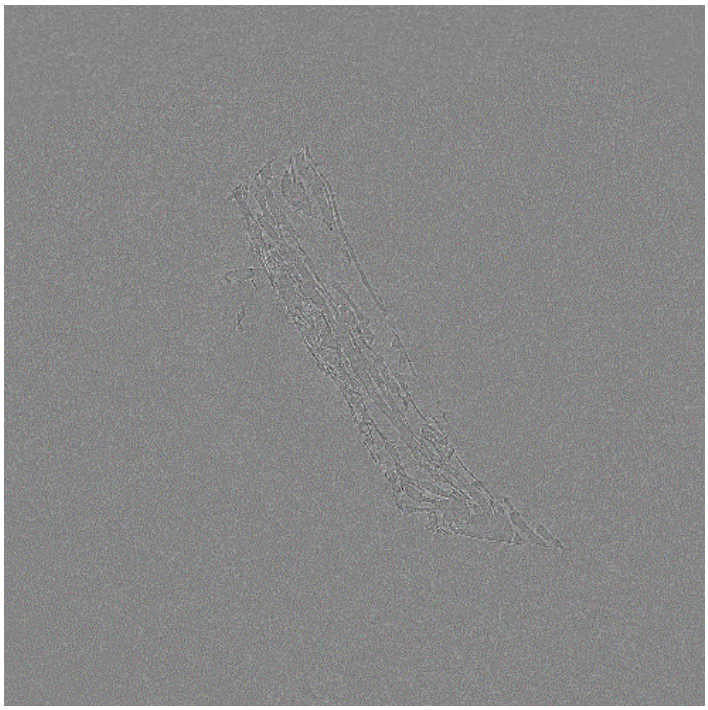
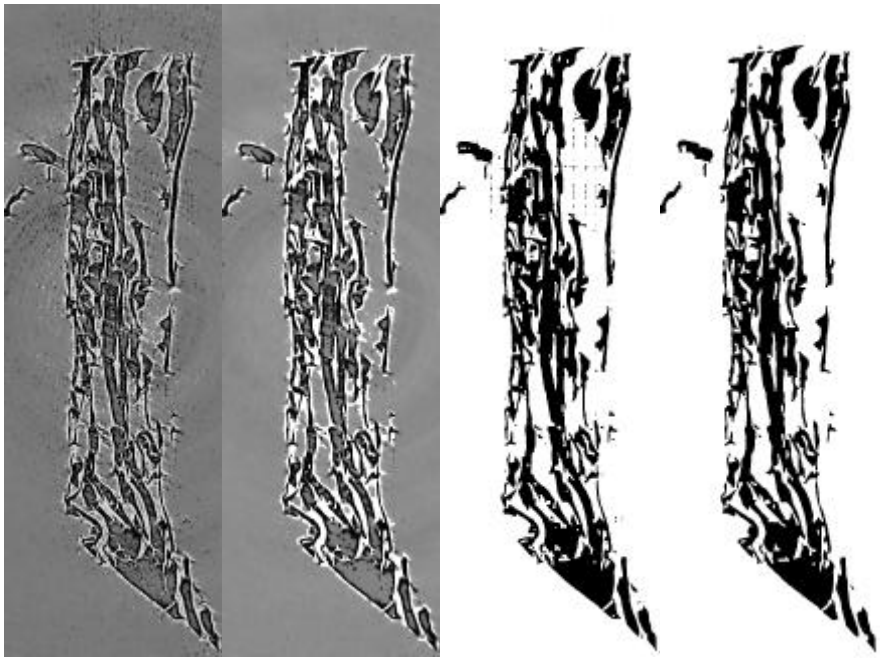


Figure 1: Reconstructed image



a **b** **c** **d**

Figure 2a-d: Procedure from reconstructed image to binary image: **a**: before closing contours, **b**: after closing contours, **c**: binary image, **d**: final binary image after noise removal.

Table 1 – Paper samples

Sample	Name	Description	State	Resolution (pixel size in µm)	ROI (pixels)
1	Black	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	dry	0,33	512 x 2048
2	KCL1	Handsheet from KCL*, 63,4 gsm, 647 kg/m^3,100% Bleached Softwood Kraft Pulp	dry	0,7	100 x 2048
3	KCL1b	Handsheet from KCL*, 63,4 gsm, 647 kg/m^3,100% Bleached Softwood Kraft Pulp	dry	0,33	512 x 2048
4	KCL2	Handsheet from KCL*, 64,3 gsm, 618 kg/m^3,90% BSKP, 10% TMP	dry	0.33	512 x 2048
5	KCL3	Handsheet from KCL*, 64,3 gsm, 564 kg/m^3,70% BSKP, 30% TMP	dry	0.33	512 x 2048
6	KCL4	Handsheet from KCL*, 63,3 gsm, 498 kg/m^3,50% BSKP, 50% TMP	dry	0.33	512 x 2048
7	KCL5	Handsheet from KCL*, 64,3 gsm, 357 kg/m^3, 100% TMP	dry	0.33	512 x 2048
8	KCL5b	Handsheet from KCL*, 64,3 gsm, 357 kg/m^3, 100% TMP	dry	0.33	512 x 2048
9	SC	SC paper, NS Saugbrugs, PM6, 56 gsm, 1206 kg/m3, 6% kraft long fibres, 12 % GW, 48 % TMP, 35 % Kaolin	dry	0,33	512 x 2048
10	Newsk	Uncal. newsprint, NS Skogn, PM3, 44,1 gsm, 403 kg/m^3	dry	0,33	512 x 2048
11	Newskb	Uncal. newsprint, NS Skogn, PM3, 44,1 gsm, 403 kg/m^3	dry	0,33	512 x 2048
12	Blackwb (Blackw failed)	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	wet (i.e. paper sample in a thin capillary filled with water)	0,33	512 x 2048
13	KCL4w	Handsheet from KCL*, 63,3 gsm, 498 kg/m^3, 50% BSKP, 50% TMP	wet	0.33	512 x 2048
14	KCL5w	Handsheet from KCL*, 64,3 gsm, 357 kg/m^3, 100% TMP	wet	0.33	512 x 2048
15	SCw	SC paper, NS Saugbrugs PM6, 56 gsm, 1206 kg/m3, 6% kraft long fibres, 12 % GW, 48 % TMP, 35 % Kaolin	wet	0,33	256 x 2048
16	Stain1 (*)	Filter paper 589[2] White ribbon, 86,7 gsm, 498 kg/m^3	Silicone oil (i.e. paper sample previously impregnated with silicone oil in a vacuum chamber)	0,33	512 x 2048

*: more complete information on KCL handsheets can be found in ref.[1].

Paper samples (continued)

Sample	Name	Description	State	Resolution (pixel size in µm)	ROI (pixels)
17	Blackt	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	Toluene (i.e. paper sample in a thin capillary filled with toluene)	0.33	512 x 2048
18	ESF	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	Dry but previously soaked very shortly in a solution of polymer dissolved in toluene	0.33	512 x 2048
19	ESG	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	Dry but previously soaked shortly in a solution of polymer dissolved in toluene	0,33	512 x 2048
20	ESH1	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	Dry but previously soaked in a solution of polymer dissolved in toluene	0,33	512 x 2048
21	ESH2 (lower conc. of polymer than in ESH1)	Filter paper 589[1] black ribbon, 76,7 gsm, 486 kg/m^3	Dry but previously soaked in a solution of polymer dissolved in toluene	0,33	512 x 2048

Appendix 1 – Beamline experimental conditions

Experiment no. ME-55 | Date/time 10/5 - 14/5 - 2000

People Ø.W. GREGERSEN, P. MYGÅRD, C. ANTOINE, R. HOLMSTAD, P.J. HOUEN, C.R., T.W. (+ local cont.)

Description: 3D PAPER STRUCTURE

Delivery mode: ☐ Uni. ☒ 2/3 ☐ 1/3 ☐ Hybrid ☐ 16-b. ☐ Single b. ☐ Other:

General optics

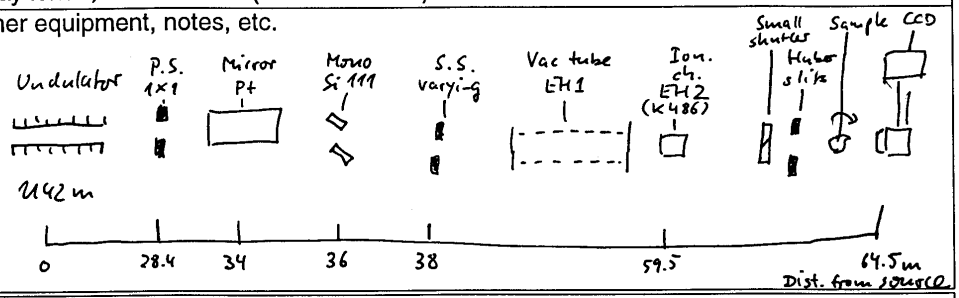
Undulator:
☒ U42m ☐ U42d Gap: 22.00 mm
Mirror:
☐ None ☐ Pd ☒ Pt ☐ Si
Deflection angle: see motor printout
Monochromator:
☐ None ☒ Si-111 ☐ Si-311 Angle: ___

Primary slits: 1×1 (H×V)
Secondary slits: 0.8 × 0.5 mm² (H×V)
Ternary slits: open wide
Absorbers: None

EH 1

Vacuum tube: ☐ No ☒ Yes
Shutter: ☒ No ☐ Yes (type: _____)
X-ray lens: ☒ No ☐ CRL (see CRL sheet) ☐ Other lens:

Other equipment, notes, etc.



EH 2

Vacuum tube: ☒ No ☐ Yes
Shutter: ☐ No ☒ Yes (type: Vincent; position: On tomo table)
X-ray lens: ☒ No ☐ CRL (see CRL sheet) ☐ Other lens:
Huber slits ☐ No ☒ Yes (position: just leave f.o.v. open)

Frelon:

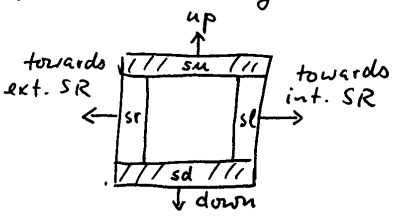
Camera: ☐ 1000 ☒ 2000
Objectives: _____
Scintillator: 3.5 µm
~~Distance to sample:~~ _____

Readout: ☐ serial ☒ mulitplex
Binning: ☒ no ☐ yes, _____
ROI: ☐ no ☒ yes, see tomo sheets
Exposure time: depends

Other equipment, notes, etc.

Ioniz. chambe in EH2 used as intensity monitor for pb2 alignment etc.

Camera field of view:



Sheet version as of December 9, 1999