New Techniques in Coating Layer Analysis: Light Microscopy and Micro Tomography

J. Kritzinger[1], U. Hirn[1], H. Schröttner[2] and W. Bauer[1]

[1] Institute for Paper, Pulp and Fiber Technology (IPZ); Graz University of Technology
RSA μSTRUCSCOP
[2] Institute for Electron Microscopy and Fine Structure Research (felmi); Graz University of Technology

Content

Review

- Coating Layer Analysis: Trends and Methods
- Scanning Electron Microscope (SEM)
- X-ray Micro Tomography (µCT)
- Serial Sectioning Technique (µSTRUCSCOP)

Comparisons

- Serial Sectioning versus SEM
- Serial Sectioning versus X-ray Micro Tomography

Summary

Coating Structure Analysis

Targets

- Coating uniformity and its influence of behavior of coated papers (printability….)
- Coating coverage and coating holdout
- Analysis of defects in the coating layer
- Surface characterization
- ….

Requirements

- Resolution in (below) the micrometer-range
- Fast analysis of large samples areas

Coating Structure Analysis

Methods used

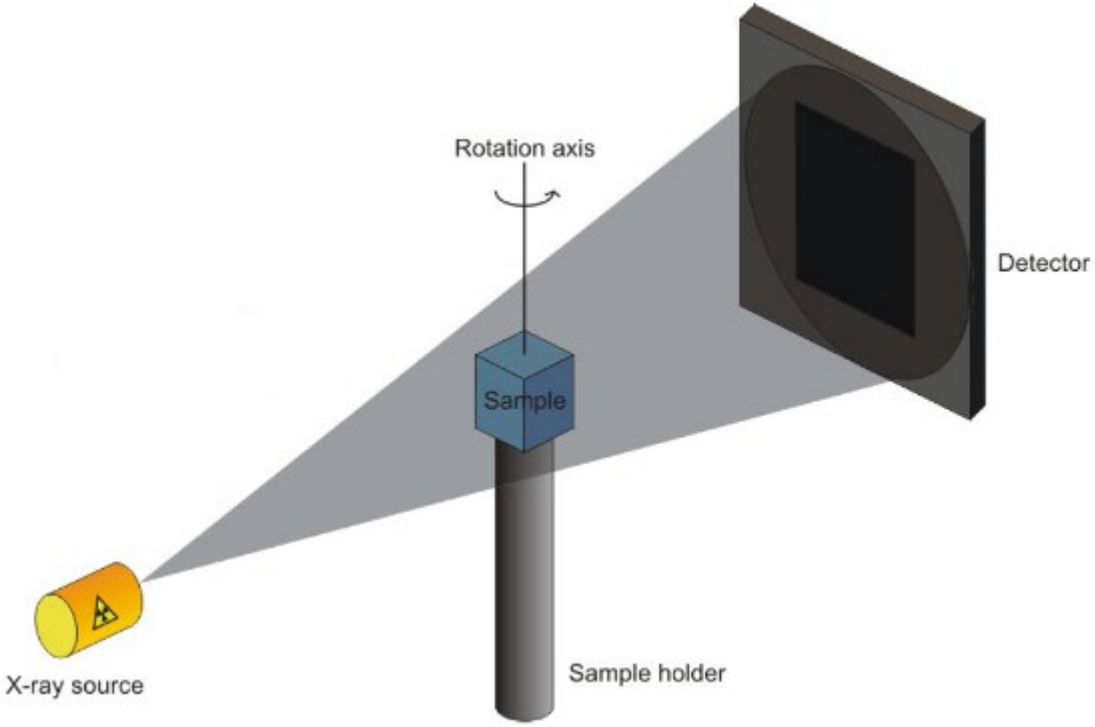
- Scanning electron microscopy (backscatter)
  - Coating thickness uniformity (Allem; JPPS, 24(10), 329-336)
  - Coating Coverage (Grön et.al.; JPPS, 27(2), 66-73)
  - Leveling / contour coat (Dahlström et.al.; 10th ACFS 2008)
- X-ray micro tomography
  - Surface characterization (Chinga et.al.; 23th PTS Coating Symposium 2007, 31.1-31.12)
  - Analysis of coating structures (Turpeinen et.al.; 22nd PTS Coating Symposium 2005, 36.1-36.14)
- Serial sectioning, light optical microscopy
  - Curtain versus blade coating (Wiltsche et.al.; 9th ACFS 2006)
  - Coating layer uniformity (Kritzinger et.al.; 10th ACFS 2008 ; 24th PTS Coating Symposium 2009)

Scanning Electron Microscopy (SEM)

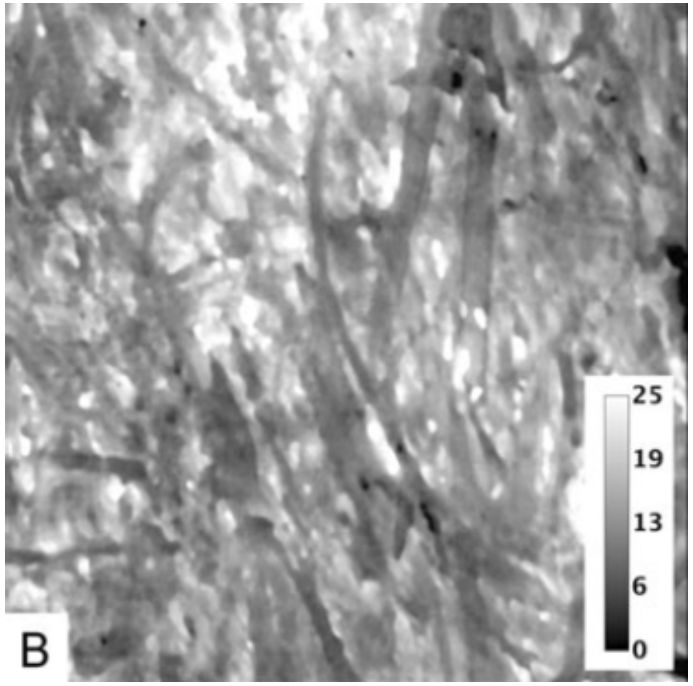
- Sample preparation: embedding in resin, sometimes staining
- Digitization of paper cross sections (2D)
  (3D approaches like serial grinding are not common)
- Resolution for coating layer analysis: wide range (~0,2 µm/Pixel)

- Application:
  - Coating structure analysis
    (coating thickness & coverage, interactions base sheet – coating)

X-ray Micro Tomography (µCT)



Sample digitization[1]



Coating thickness map[2]

[1] Turpeinen et.al..; 22nd PTS Coating Symposium 2005, 36.1-36.14

[2] Chinga-Carrasco et.al..; Journal of Microscopy; 232(2), 212-224

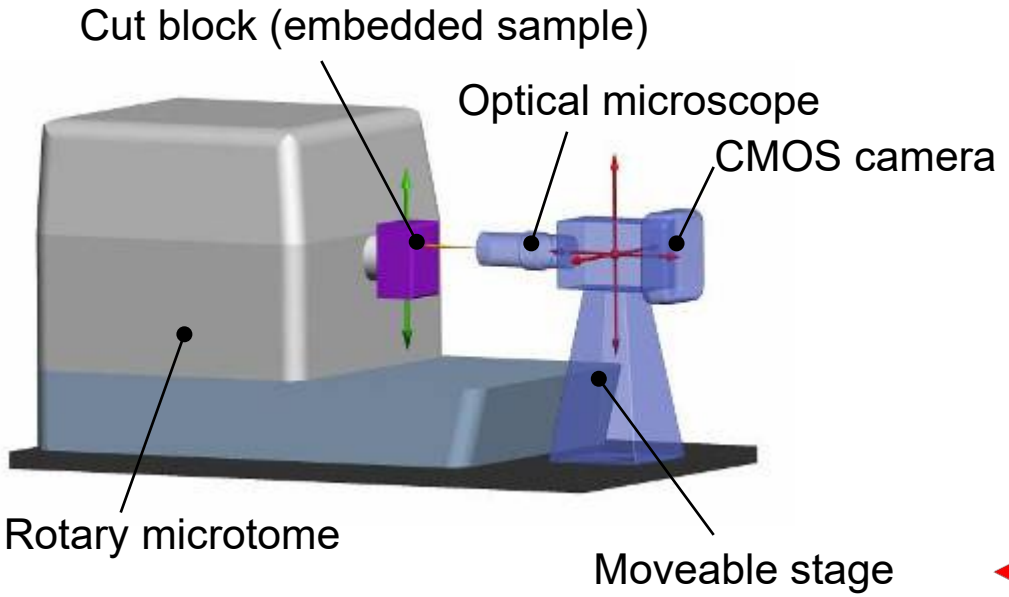
X-ray Micro Tomography (µCT)

Desktop scanners most common.

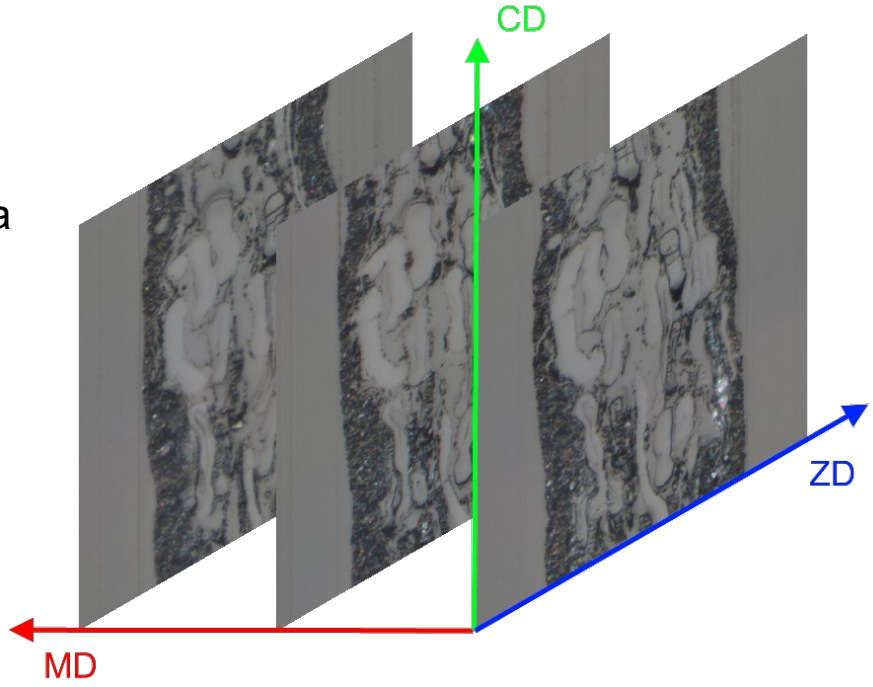
- Sample preparation: not required (cutting)
- Accessibility to 3D-data sets 2 x 2 mm²
  (the higher the resolution, the lower the sample size)
- Resolution for coating layer analysis: 0.86 µm/Voxel

- Application:
  - Analysis of coating structures
    (base paper and final paper topography, coating thickness …)
  - 3D reconstructions of fibrous webs
    (textiles, press felts,…)

Serial Sectioning Technique (µSTRUCSCOP)



Digitization technique[1] (µSTRUCSCOP)



3D-data set

[1] Wiltsche et.al.; Transaction of 13th Fundamental Research Symposium 2005, 853-899

Serial Sectioning Technique (µSTRUCSCOP)

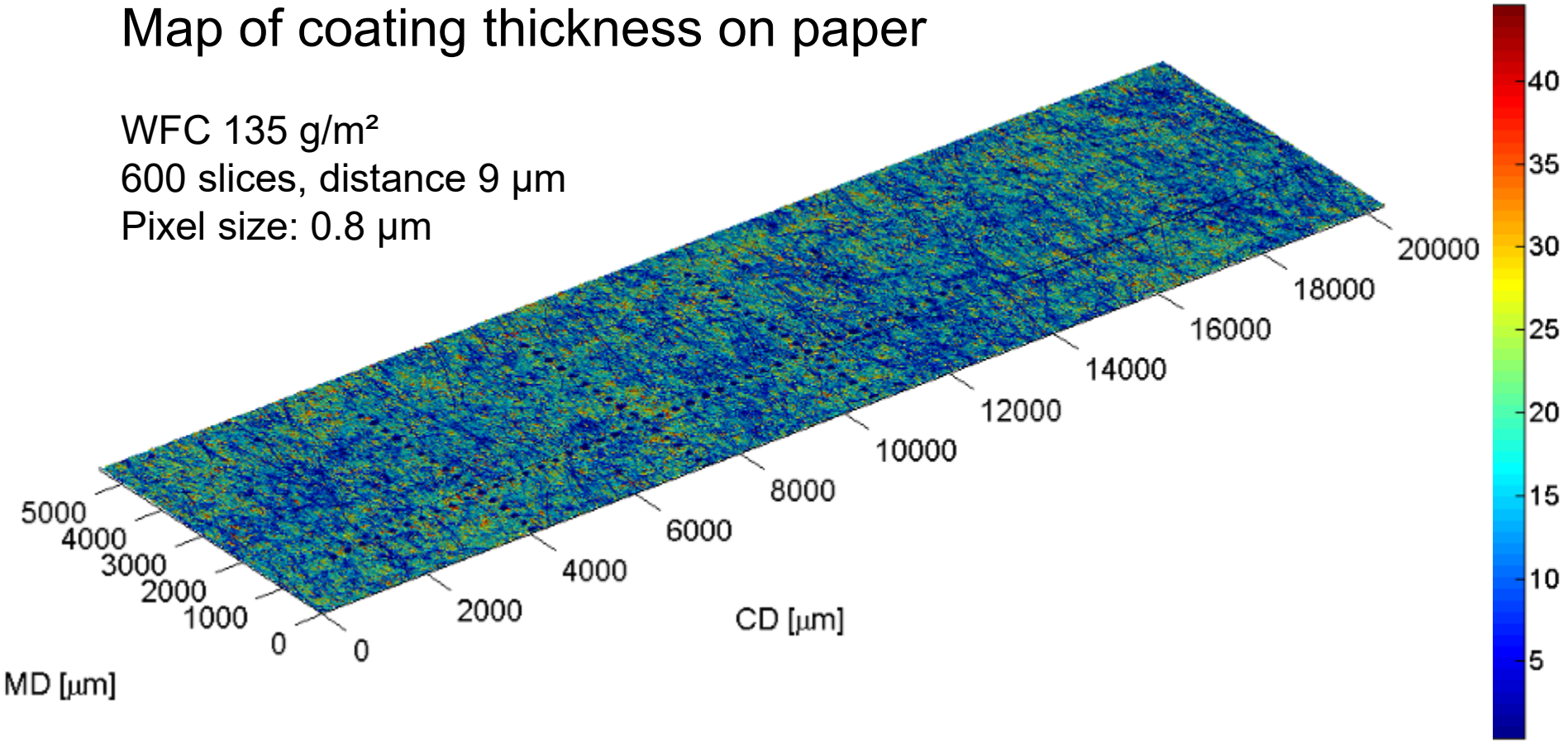
- Sample preparation: embedding in resin
- Accessibility of large 3D-data sets (>1 cm²)
- Resolution for coating layer analysis:
  - Image plane: 0.8 – 0.4 µm/Pixel
  - Slice thickness: 2 – 12 µm
  - Slice length: >20 mm

- Application:
  - Representative analysis of coating structures
    (coating thickness & coverage, interactions base sheet – coating)
  - Coating thickness maps
    (correlation coating thickness – printing result)

Serial Sectioning Technique (µSTRUCSCOP)

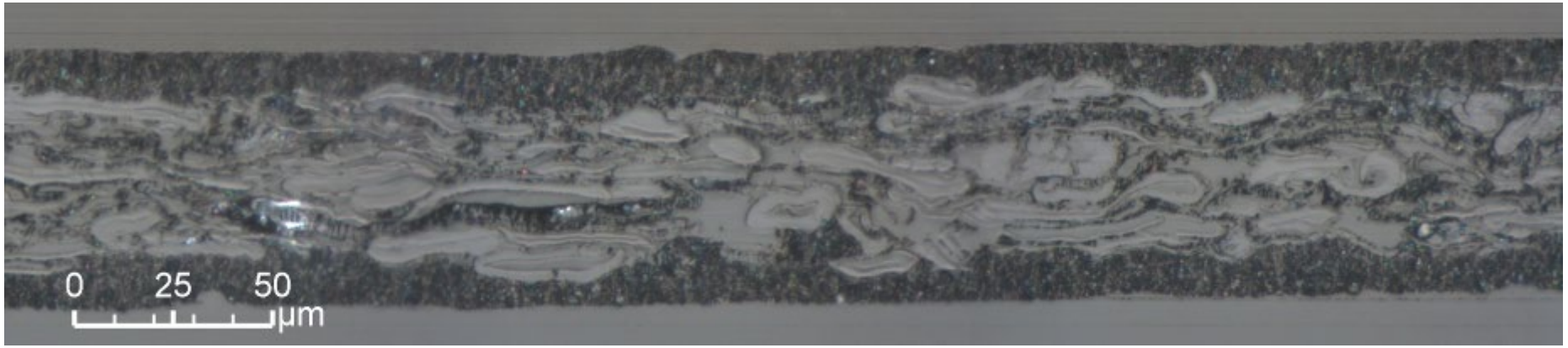
Map of coating thickness on paper

WFC 135 g/m²
600 slices, distance 9 µm
Pixel size: 0.8 µm

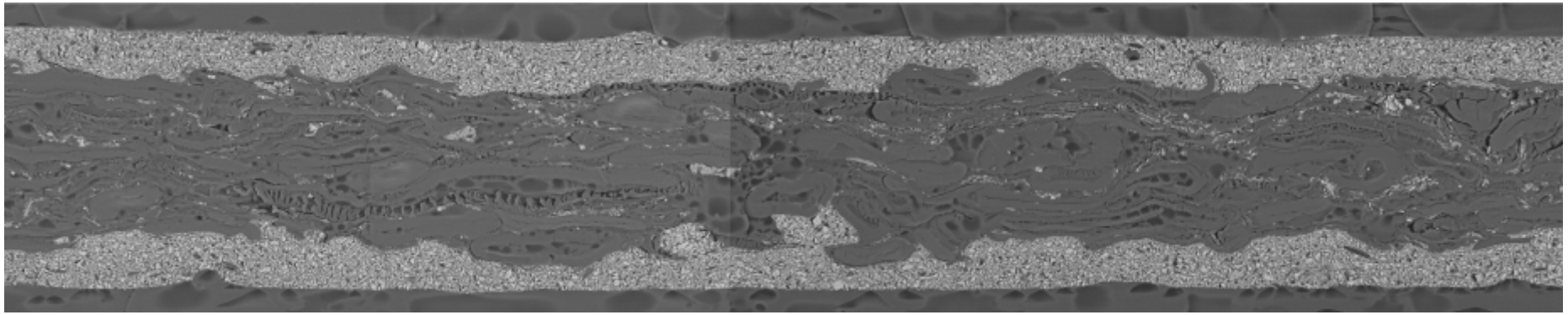


Hirn et.al.;Transaction of 14th Fundamental Research Symposium 2009

Serial Sectioning versus SEM

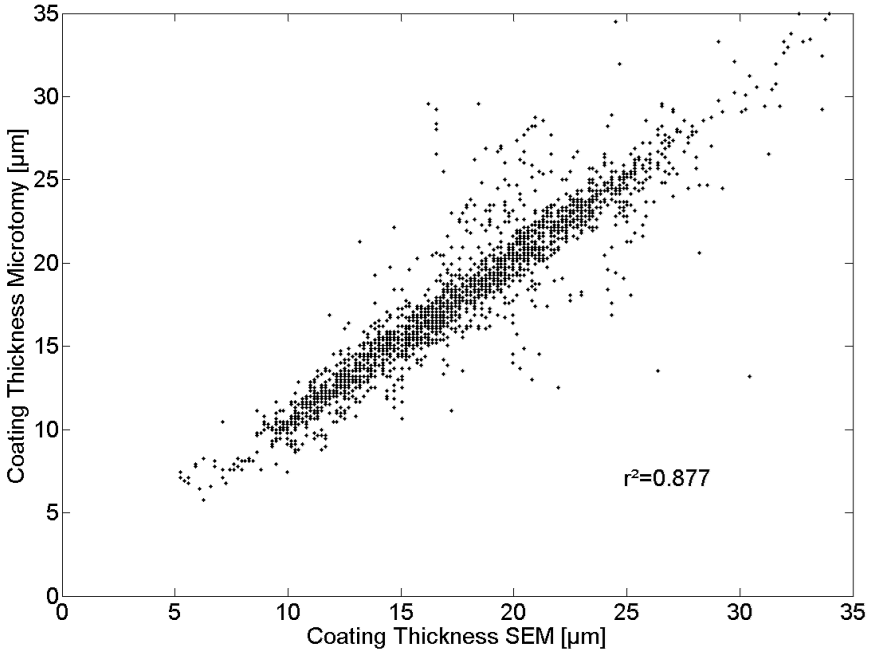


Serial Sectioning IPZ-system (µSTRUCSCOP), pixel size 0.16 µm



SEM (felmi), pixel size 0.0558 µm

Serial Sectioning versus SEM



- Strong correlation of coating thickness data obtained from SEM and serial sectioning images (at the same position).

Serial Sectioning versus SEM

- 2D-analysis: similar results in coating layer analysis

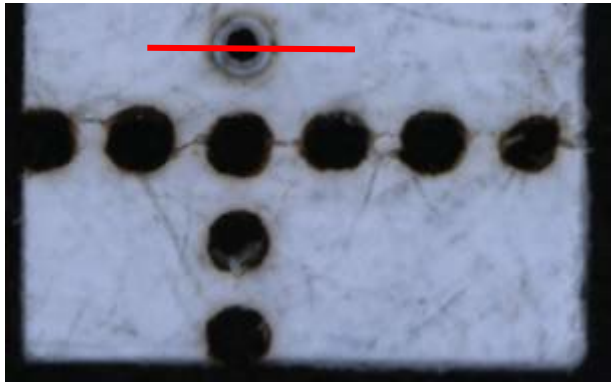
SEM

- Very high resolution
  → Details (pigments, pores) in the coating layer are visible
- Coating layer analysis on single cross sections

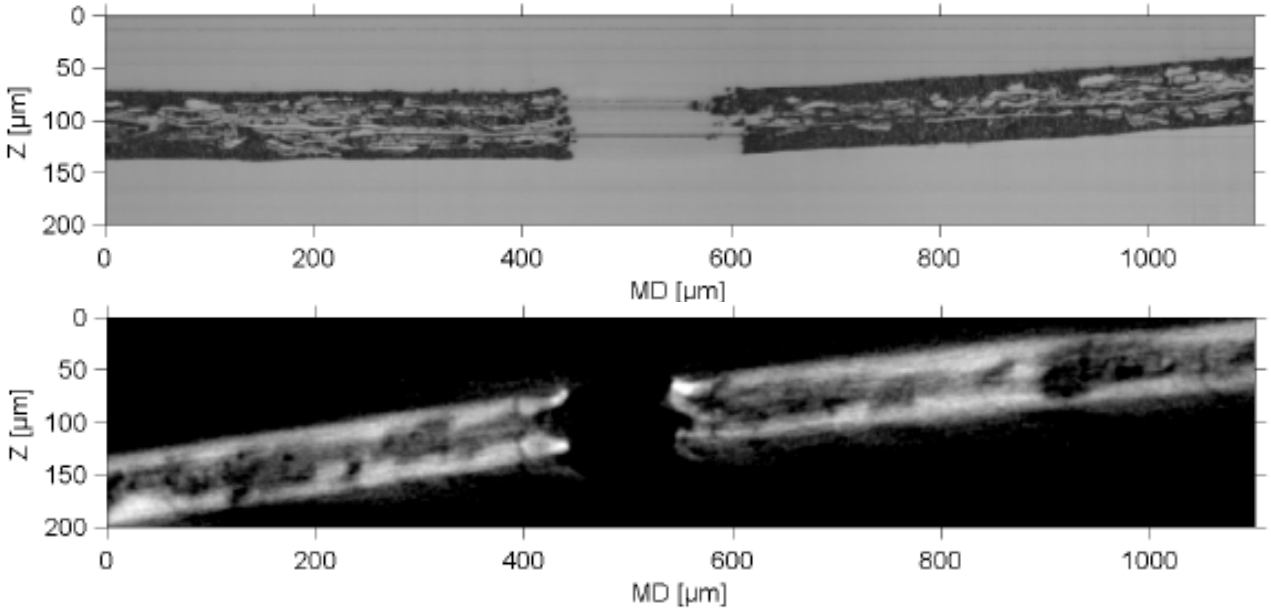
Serial Sectioning

- Surface irregularities are visible
- Coating base paper interface sometimes not very clear (better at lower resolutions)

Serial Sectioning versus Micro Tomography



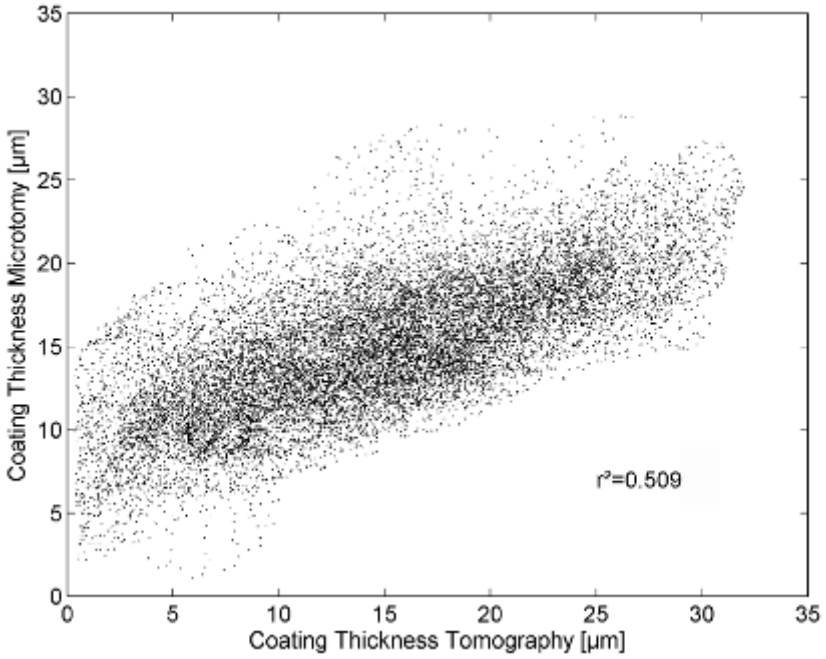
Serial Sectioning (µSTRUCSCOP), pixel size 0.83 µm



Micro Tomography Skyscan1172, voxel size 1.37 µm

Hirn et.al.; Transaction of 14th Fundamental Research Symposium 2009

Serial Sectioning versus Micro Tomography



- Limited correlation of coating thickness data obtained from X-ray micro tomography and serial sectioning
- Trend to overestimate coating thickness with micro tomography

Serial Sectioning versus Micro Tomography

- 3D analysis: variations in the obtained results

X-ray micro tomography

- Detection of fine structures in the coating layer is difficult (threshold level, noise,…)
- Non-destructive measurement

Serial Sectioning

- 3D data set almost free of artifacts
- No relation between sample size and resolution

Summary

	Resolution	Image Quality	Sample Size 2D	Sample Size 3D	Time Effort	Remarks
SEM	+	+o	+	–	–	well established Technique
µCT	–	–	–o	o	+	rapid development
µSTRUCSCOP	o	+o	+	+	o	sample size

➔ All methods are suited for coating structure analysis