## Application of a Novel Automated Serial Sectioning Technique in the 3D Analysis of Paper Structures

J. Kritzinger<sup>1</sup>, M. Wiltsche<sup>2</sup>, M. Donoser<sup>3</sup>, and W. Bauer<sup>1</sup>

 RSA µSTRUCSCOP, Institute for Paper, Pulp and Fiber Technology, Graz University of Technology, Kopernikusgasse 24/II, A-8010 Graz
Andritz AG, Stattegger Strasse 18, A-8045Graz
Institute for Computer Graphics and Vision, Graz University of Technology, Infelddgasse 16/II, A-8010 Graz

## johannes.kritzinger@tugraz.at

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Especially for highly heterogeneous materials such as paper knowledge of the 3D structure is a key to better understand the material properties. Given the dimensions of the main papermaking raw materials ranging from below one micrometer to a few millimetres the method used in the evaluation of the 3D structure has to fulfill two severely conflicting requirements. First, a high spatial resolution at least in the range of 0.5 to 1  $\mu$ m is necessary to be able to identify important structure details. Second, a sufficient sample size of close to one square centimeter (for a planar material such as paper) is required to obtain reliable and statistically meaningful results.

Since methods like SEM serial sectioning or microtomography are not yet able to fulfill these requirements, we have developed an episcopic serial sectioning technique which allows the digitization of large flat samples, like e.g. paper, almost independently from the chosen resolution [1]. We believe that the method is also of interest for other industrial materials such as e.g. textiles, non-wovens, fiber reinforced polymers and other composites. The technique combines automated microtomy and light optical microscopy. The microscope is fixed on a movable stage and is mounted in front of the microtome; see Figure 1. The image acquisition procedure is fully automated. Repeatedly, thin slices are cut off with a diamond knife from a resin embedded sample. After each cut sequence, the surface of the cut block is scanned. To increase the field of view, a series of subsequent images is taken. This procedure provides a digital 3D representation of the material investigated, see Figure 2. The voxel size of the representation is defined by the thickness of one slice (distance between the image planes) and the pixel size in the image plane.

The analysis of paper samples is usually carried out at slice thicknesses of 1 to 10  $\mu$ m and an optical resolution of 1.1 to 0.6  $\mu$ m. Up to 20 mm long paper cross section are analyzed and depending on the purpose of the analysis up to 1000 cross sections are investigated.

The obtained paper cross sections are further processed using newly developed image analysis routines, e.g. [2] for the detection of fiber cross sections of individual wood pulp fibers. The detected fiber cross sections are used to digitally reconstruct the fiber network in a sheet of paper, see Figure 3. The knowledge of the spatial location of the wood pulp fibers also allows the evaluation of important pulp fiber properties such as fiber wall thickness or lumen area, which are difficult to asses with other methods such as CLSM or SEM.

A further application is the analysis of a pigment coating layer, which is applied to the paper surface to enhance the optical properties as well as printability of paper. Figure 4 shows a map of local coating thickness and is an example of the potential of our method to measure larger samples. The sample area is 110 mm<sup>2</sup> and 600 paper cross sections (slice thickness 9  $\mu$ m) with a length of more than 20 mm have been investigated automatically.

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**Figure 1.** Schematic diagram of the prototype built. The microscope is fixed on a movable stage in front of the microtome. [1]

**Figure 2.** Digitization results in a sequence of images. Here coated paper cross sections are shown exemplarily.



**Figure 3.** Digital reconstructed fiber segments in a paper network. The sources of this reconstruction are fiber cross sections of individual fibers, which were detected with an image analysis algorithm along the digitized paper cross section sequence.



**Figure 4.** Map of local coating thicknesses on paper. The sample covers an area of 110 mm<sup>2</sup>. 600 paper cross sections (slice thickness 9  $\mu$ m) with a length of more than 20 mm have been investigated. The line below 2000  $\mu$ m in MD results from image analysis artifacts. The holes are paper perforations.