Applied Microscopy for the Paper Industry

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Papermaking technology spans the sciences of biology, chemistry, engineering, and physics and often requires process understanding only possible through microscopy. Biotechnology is now applied to improve tree reproduction, disease resistance, and wood quality. Pulp and paper mills alter the physical and chemical properties of fiber, and involve energy intensive prœesses that can result in corrosion, scaling, and paper contamination. Coating and printing of paper and board demand additional material performance properties that are developed at the microscopic level. Paper product development, production, and quality control have all benefited from microscopic analysis.

Various forms of compound light microscopy and stereoscope imaging are essential to paper evaluation laboratories. Identification of pulp types and fiber species is accomplished by light microscopy using anatomic features with the aid of specialized stains. Microfibril angle in wood fibers can be determined with DIC microscopy and image analysis [1]. Contaminant particles, off color spots, and other appearance defects are investigated with a combined approach. This typically begins with light microscopy and may require FTIR microscopy or SEM/EDS. Sources of contamination can include additive packaging, improperly screened raw materials, and environmental dusts. Deposits from components of virgin or recycled fiber can build up on rolls and roll fabrics in the natural course of mill operation. Printing plants have their own picking and deposit problems that vary with types of paper and printing processes.

SEM and TEM both have extensive applications in pulp and paper science [2]. SEM is more commonly used than TEM due to the nature of paper industry needs and, perhaps, the ease of sample preparation and instrument operation. SEM images of paper surfaces may reveal the degree of fiber bonding and the amount of external fibrillation, a consequence of the pulping, refining, and sheet forming processes. Tissue papers are formed with high bulk and porosity, and a controlled fiber furnish gradient, to optimize tactile smoothness and absorbency. Offiæ copying and printing papers are heavily loaded with mineral fillers for a flatter surface with higher brightness. The distribution of fillers at the surfaces or in cross-section can be mapped with backscattered electron imaging. EDS is used to obtain elemental spectra that, along with morphology, often delineate filler types. At higher magnifications, polymeric sizing agents and coating binders can be visualized by TEM.

Cross sections of paper and board expose important thickness (Z) dimensional properties. These include the density and porosity of the sheet, the thickness and uniformity of coatings, and the retention of filler. Microtome sections for light microscopy are prepared to determine the penetration of starch sizing. For SEM examination of large sections, ground and polished cross sections have become more common [3]. The composition and microstructure of coatings can be determined in this way. The collapse of fiber walls and, sometimes, internal fibrillation are revealed. Figure 1 shows a polished cross section of a lightweight coated paper.

The availability of variable pressure and environmental scanning electron microscopes (VPSEM, ESEM) has made possible the study of paper fiber with its natural moisture content, and the

evaluation of drying effects [4]. Coating components, like latex and plastic pigment, can be imaged without damage [5]. Additional applications are investigations of wood structure and mill corrosion products without the need for conductive coatings.

Operational problems in pulp and paper mills are sometimes resolved with the aid of SEM. Scales in pipes or vessels can develop in the evaporators of a pulp mill or the bleach plant of a paper mill. Shutdowns for maintenance cause expensive interruptions in production Light microscopy, SEM/EDS, and x-ray diffraction are commonly used to determine the texture and composition of mill scales. With this information, mill operations can be modified to minimize scale formation and expedite removal. Pulping liquors used in the kraft process may contain solids derived from feed chemicals, wood chips, or corrosion. Black liquor from the recovery boiler or green liquor from the washing of boiler residue contains fine solids that can be measured and chemically analyzed by SEM/EDS. The texture of fume particles that are produced in recovery boilers, as well as chars that are generated in black liquor gasification, have been measured by SEM to understand the impact of adjusting control variables. [6]

References:

- [1] G.F. Peter et al., J. Pulp & Paper Sci., 28 (2002) in press.
- [2] D.R. Rothbard, in Z.R. Li (ed.), *Industrial Applications of Electron Microscopy*, Marcel Dekker, New York, (2002) in press.
- [3] G.J. Williams and J.G. Drummond, J. Pulp & Paper Sci., 26 (2000) 188.
- [4] T. Enomae and P. LePoutre, Nordic Pulp & Paper Res. J., 13 (1998) 280.
- [5] Y. Xiang and D.W. Bousfield, J. Pulp & Paper Sci., 26 (2000) 221.
- [6] This work was made possible by the support of the member companies of IPST.

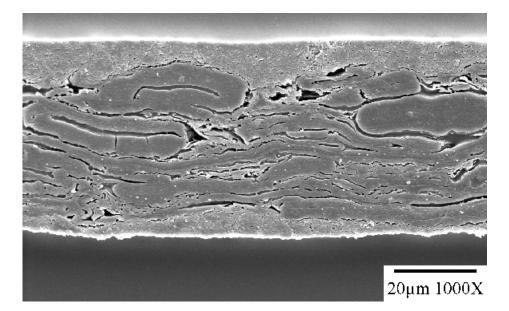


Figure 1. SEM secondary electron image of polished and etched cross section of lightweight coated paper. Bar = $20 \,\mu$ m. (Photo by G. Maghiari, IPST. Reproduced from [2] by courtesy of Marcel Dekker, Inc., www.dekker.com)