

Determining paper-coating thickness with electron microscopy and image analysis

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ABSTRACT: For several decades, the determination of a coating thickness was a tedious and time-consuming task because of the uneven nature of the base sheet. The use of image-analysis techniques has allowed the determination of average coating thicknesses for individual samples up to several hundred micrometers in length. Statistical data have shown acceptable precision with standard deviations used to estimate base paper roughness.

KEYWORDS: Coating weight, electron microscopy, image analysis, measurement, paper, performance evaluation, thickness.

Production economics and various final performance aspects of coated paper can be related to the amount of coating present on a given sheet. In turn, the ability to measure the amount of coating present is of great importance in interpreting these factors. The amount of coating present can be measured in terms of coating weight, coating thickness, and their relationship—coating density. Of these measures, coating thickness is generally the most readily attained from individual paper samples (1, 2).

Historically, coating thickness has been examined using paper cross sections and light microscopy, with the earliest use of paper cross-sectioning reported early in this century (3, 4). While different techniques abound, the

embedding and subsequent cross-sectioning of paper is still a useful technique to characterize the internal structure of a piece of paper (5).

Many light-microscopy techniques, such as those using transmitted, polarized, or reflected light, have been used to observe various characteristics of the sheet. These techniques were used to study pigment/filler distribution in the Z direction, sheet formation, starch distribution, and various defects in the sheet. In the recent past, scanning electron microscopy (SEM) has been added as a tool to characterize paper cross sections. SEM and the complementary X-ray technique, energy dispersive spectroscopy (EDS), can do much of this work as well. Now seen as complementary

techniques, both light and electron microscopy have also benefited from the increased use of computerization (6, 7).

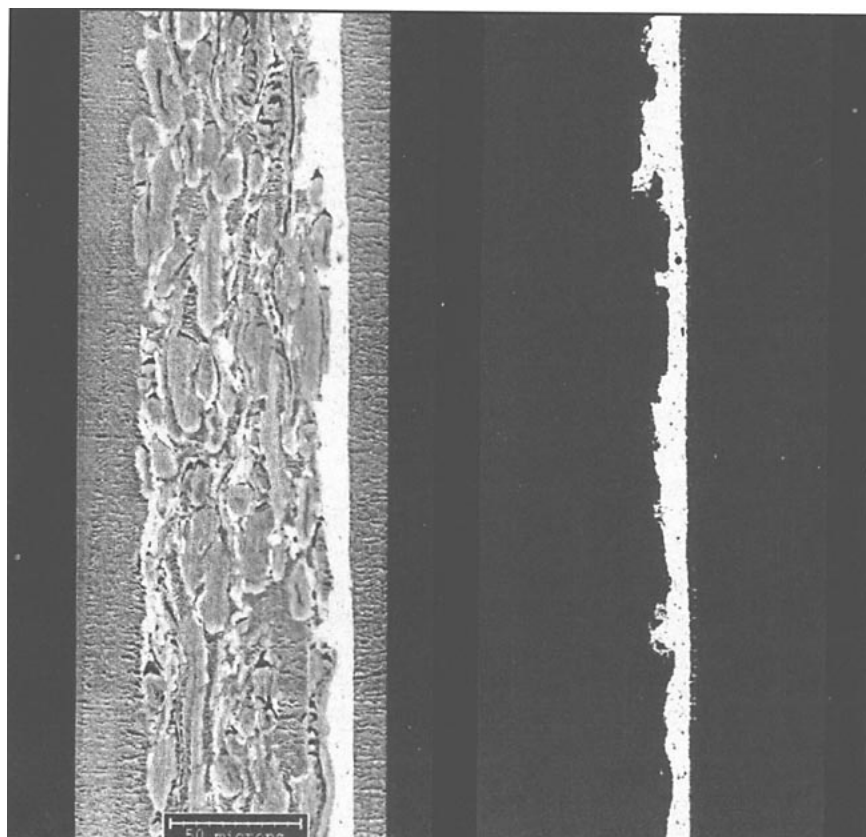
Measurement of a coating thickness can be completed similarly with both imaging techniques. Basically, coating thickness was measured manually across the coating, and a relative estimate was recorded. The nature of paper dictated that a relative estimate be taken due to a typically rough base sheet and the resulting variation in coating thickness. Any effort to secure a representative coating thickness value required a multitude of points to be measured manually (1, 2).

Using digitally acquired cross-sectional images of paper, an image-analysis system can be used to calculate the average coating thickness along a given sample length. These images can be used to isolate a paper coating from the sheet based on brightness and contrast resulting from atomic weight differences using back-scattered electrons (6). The isolated paper coating area can then be used to calculate the average thickness with measured total image area and sample length. Acquiring several images along a given sample results in a good representation of thickness variations due to the natural formation and other characteristics of the sheet. The statistical data formulated can then be used to characterize base sheet roughness using the calculated standard deviation.

Precision, as expressed by the standard deviation, was acceptable. It was shown to be related to base sheet

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4. Typical 256 gray-level image and its isolated coating



I. Thickness values

	<i>T</i>	<i>S</i>	<i>RSD</i>	<i>N</i>
Grade A, MD & CD	9.52u	1.72	18.1%	60
MD	9.40	1.49	15.9	30
CD	9.60	1.94	20.2	30
Grade B, MD & CD	5.64	1.19	21.0	60
MD	5.73	1.20	21.0	30
CD	5.57	1.19	21.0	30
Grade C, MD & CD	10.61	1.81	17.1	60
MD	10.27	1.49	14.5	30
CD	10.94	1.58	14.4	30
Grade A, 2 Sigma	9.53	1.44	15.1	56
Grade B, 2 Sigma	5.41	0.79	14.6	56
Grade C, 2 Sigma	10.38	1.53	14.7	57

roughness. Data for coating thickness taken from three different paper samples gave accuracies over 90% using confidence limits of one standard deviation.

Experimental image analysis

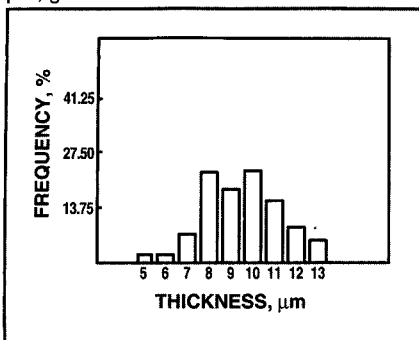
Paper samples for the statistical analysis of the method were selected from three different commercially available papers. Common embedding techniques were used with hand-cut

samples placed in a 7 mm x 20 mm capsule for embedding. Each sample was cut to a width of 2 mm to 3 mm. The embedding medium was an epoxy resin with good penetrating characteristics, cured at 75°C for 48 h to harden the medium. After hardening, each sample was cut using a glass knife and ultramicrotome. After Au-Pd sputtering, the samples were submitted to the SEM, a JEOL T330. Images were acquired using a Princeton Gamma Tech IMIX System at magnifications between 150X and 300X. Proper magnification depended on coating thickness to ensure sufficient resolution.

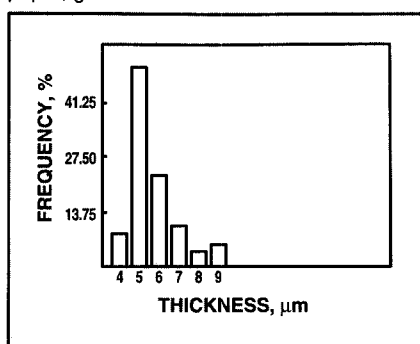
Imaging was accomplished using backscatter electrons to facilitate the differentiation between the coating and the base sheet, as well as the embedding medium. Backscattered electrons resulted from the interaction between the primary electrons and the material being imaged. The resulting energy of backscattered electrons was directly related to atomic weight of the target material. Therefore image contrast and brightness were also directly related to the element present. Elements mainly present in the coating, such as calcium, silicon, aluminum, and titanium, could be imaged much more brightly than the surrounding high-carbon-content materials, thus allowing all inorganic material to be clearly distinguished from the rest of the sheet (6).

After image acquisition, the coating area was separated from the image. With image contrast, a function of atomic weight, gray levels could be used to differentiate clearly between high carbon content areas, such as fibers and the embedding medium, and the paper coating. The area of the coating was then obtained based on the number of pixels within the coating gray-level range, which was determined by the brightness and contrast of the coating. Image pixels are the individual points which make up the image, each having a certain gray level. Image resolution is ultimately based on the number of pixels within a given image. The images used here were 1024

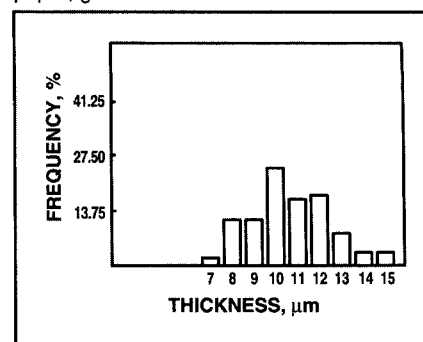
1. Coating thickness distributions—coated paper, grade A



2. Coating thickness distributions—coated paper, grade B



3. Coating thickness distributions—coated paper, grade C



x 1024 pixel images. A total of 256 gray levels were used for brightness and contrast differentiations. The gray-level range used for the coating varied between 10 and 50 individual levels or shades of gray. The coating was separated from the sheet digitally to remove any possible interference from internal pigment/filler present in the sheet, which would also have this same gray-level range.

Total image area was calculated in μm^2 , using the measured dimensions of the image. Using the predetermined gray-level range for the coating, the percent of the total image area was obtained for this range by means of the image analysis software. This is simply the percent of total-image pixels in the assigned gray-level range for the coating. The sample was positioned so that either the X or Y dimension of the image equaled the sample length. Using the equation for area, it was possible to determine the average width of the coating, or coating thickness, for the entire sample length. At a typical magnification of 200X, sample length was measured at 300 μm for these measures. **Figure 1** shows the resulting isolated coating segment. The selected gray-level range has been used to isolate the coating by combining the original 256 gray level into two gray levels—one level for the coating and one for the remaining area.

Because of the natural formation changes in paper and the relatively small sample length, several samples were obtained from each 8 in. x 10 in. sample. Ten samples were cut in ma-

chine direction and ten in cross-machine direction. From each of these 2 mm to 3 mm wide samples, three separate 300 μm lengths were imaged and the coating thickness calculated for each. This resulted in 60 coating thickness values from the original sample. This process was repeated for the three types of paper.

Discussion

The technique used in the experiment appears to be a quick method for determining a good representation of coating thickness and base-sheet roughness. The method demands instrumentation that is already available in many laboratories. An experienced user can determine an average coating thickness measurement from four acquired images in about ten minutes outside sample preparation. Sample preparation would vary with embedding and imaging techniques, but these techniques are readily applicable to multiple sampling methods to increase sample throughput (7).

Statistical analysis of data from the three sheets was completed to give some idea of the minimum number of samples required to report a good representation of the true coating thickness. Total population data were used to calculate the coating thickness, T , and standard deviation, S , that was then assumed to be true. **Table I** shows the resulting values for the three samples. Also included in this table are values calculated from data that were within two S of the original calculated

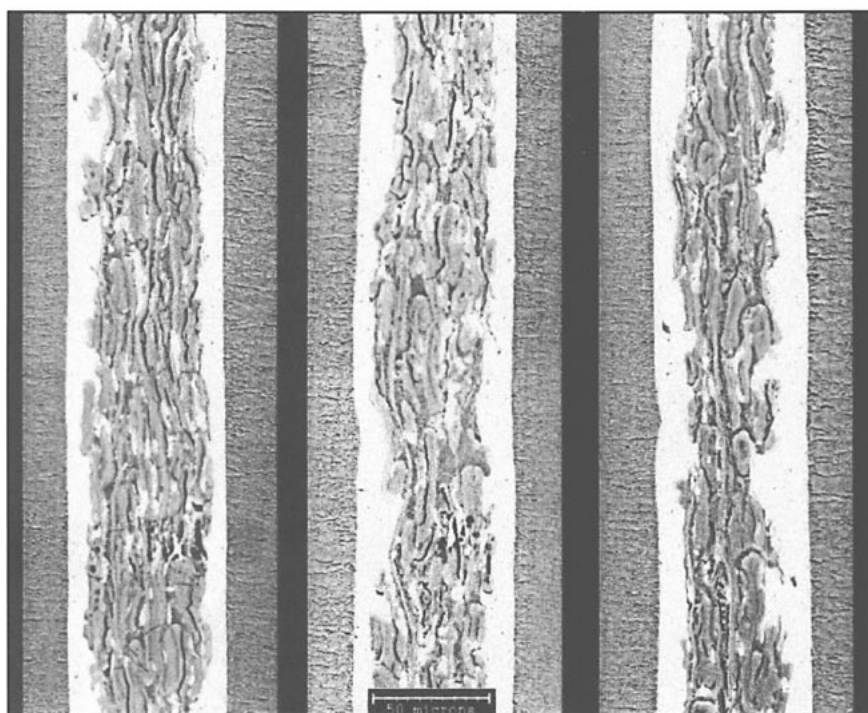
average. The resulting relative standard deviation values were remarkably similar among these samples. **Figures 2–4** indicate the distribution of thickness values for each sample.

Sample sets of 3, 4, 5, and 6 measures were used to calculate average coating thickness values, t , and standard deviations, s , which were then compared with the total population data. These values, taken randomly from the original samples, were obtained to determine the minimum number of measures required to reproduce averages within one S of the original average. The measures used were each taken from a separate sample randomly selected. These comparisons indicated 90% of calculated t values were within one S of T in all sample sets of at least four measures.

The calculated standard deviation, s , for the sample sets did not reproduce S well and were usually higher by 20%. This result would be expected based on the small population size. Comparisons between samples are thought to be still valid as long as the number of measures is the same.

The precision of this method for calculating coating thickness from a typical image for different users was extremely high, with a relative standard deviation of less than 5%. This precision appears to be a function of the location and amount of internal pigments and fillers present in the sheet. The imaging technique used does not differentiate among clays, calcium carbonate, or other inorganic fillers and pigments used internally or in

5. Three combined cross sections from a grade with few isolated surface fibers and variation in base sheet thickness



II. Thickness values for samples in Fig. 6

	<i>T</i>	<i>S</i>	<i>RSD</i>
Sample 1	8.36	1.78	21%
Sample 2	7.37	0.99	13%

the coating. For some highly filled sheets with an open surface allowing deep coating penetration, the interface between coating and filler is not readily apparent and must be estimated. However, this type of occurrence is rare.

The use of a glass knife for cutting the cross sections also influences sampling error. Surface chatter on the cross-sectioned surface caused by the knife can lead to edge effects in the image which may interfere in coating area determination. This occurs in very

thin high-carbon-content areas which tend to charge and cause artificial areas of high brightness and contrast. These areas may match the gray-level range of the coating, which would alter true coating areas (6). In addition, the particles in the coating itself may not cut smoothly, creating a limited number of very small ($<0.5\ \mu\text{m}$), relatively deep pockets in the sectioned coating. These pockets act as energy sinks, leading to a loss of pixel area in the selected gray-level range. The use of a diamond knife would be expected to eliminate these knife effects.

The calculated standard deviations for coating thickness would appear to be an excellent indication of base-sheet roughness. Due to the leveling effect of most paper coatings, the naturally rough surface of the base sheet is smoothed considerably when coated. This, combined with the relatively smooth coating surface evident in most coated papers from cross-sectional analysis, would indicate that any variation in measured coating thickness values is directly caused by base sheet variation. The variation in coating

thickness, as measured by the standard deviation of this technique, would then provide a measure of base sheet surface variation or roughness. Some extremely rough matte papers or papers with a severe contour coating would introduce an additional variation in standard deviation from actual surface variation in the coating.

The sample length used to calculate thickness measures will affect this relationship. They should not become too large. In the examples provided in Figs. 5 and 6, two sheets are presented with varying calculated coating thickness values and standard deviations. The first sheet appears to be a supercalendered sheet with few isolated surface fibers, while the second sheet appears not to be supercalendered with numerous isolated surface fibers. From statistical data using $300\ \mu\text{m}$ image lengths, the first sample was shown to have a rougher base sheet mainly due to variations in base sheet caliper. Measured thickness values are included in Table II. The base sheet of the second sample might appear to be rougher, but on a smaller sample length scale due to the number of isolated fibers. The standard deviation of the coating thickness is lower for the second sample due to the averaging effect over $300\ \mu\text{m}$ and the consistent caliper of the base sheet.

While these techniques rely on SEM-acquired images, light microscopy could also be utilized if the proper combination of optical and chemical staining can be achieved. Many image-analysis systems can acquire images from a video system, and most of the same software can be used for these systems.

Conclusion

Much of this work builds on methods that have been studied and refined for many years. The method described here is yet another utilization of the increasingly affordable computer power available today. The technique allows the determination of paper-coat-

Coating Thickness

ing thickness as well as a reasonable measure of base-sheet roughness. Using skills already developed for traditional paper characterizations, this method easily expands the use of paper cross sections. $\square$

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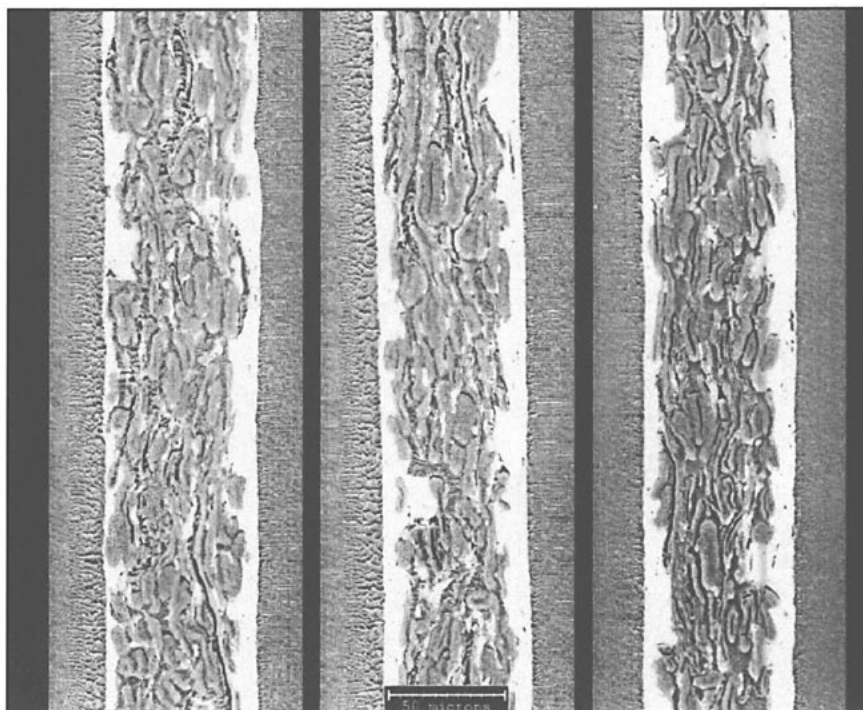
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6. Three combined cross sections from a grade with numerous isolated surface fibers but consistent base sheet thickness



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