Soft X-ray imaging can be used to assess sheet formation and quality

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ABSTRACT: *Low-energy (<10 keV) x-ray radiography can be used to assess formation with a spatial resolution on the order of one fiber diameter. Digital image processing can be used to quantify mass and floc size distribution through histogram analysis and two-dimensional Fast Fourier Transforms, respectively. Superior speed and resolution may be achieved via optical image processing techniques. In addition, formation can be assessed independently of sheet color, stickies and other contaminants are easily quantified, and the quality of internal layers in multilayer sheets can be assessed.*

KEYWORDS
Analysis
Formation
Radiography
X-ray spectroscopy

A universally accepted definition of sheet formation and a fundamentally sound technique for its measurement on some absolute basis continue to be basic needs of the paper industry. For the purposes of this work, we define formation as simply the in-plane mass or basis weight distribution. Ideally, one would like to quantify not only the statistical variation of basis weight, given some described spatial resolution, but also the spatial variation of mass, or floc size distribution. An easily applied technique to determine true sheet mass distribution with fine spatial resolution would be a powerful tool for assessing sheet quality, as it would allow the comparison of products made under vastly different conditions on an absolute scale.

Current approaches fail to realize the objective of readily determined mass distribution on the fine scale. All optically based approaches rely on a scattering mechanism, which is not a true measure of mass and can be sensitive to the presence of fillers and colorants. Also, these approaches become more difficult to apply as basis weight increases. While the various optical schemes can be used to qualitatively rank sheets which are not vastly different, they fail to provide a true measure of density distribution, and the different approaches have been shown to disagree in their rankings of identical sheets. Finally, the resolution of such devices is quite limited.

Therefore, there exists a need for some technique for formation measurement that meets the following criteria:

1. A true measure of in-plane mass distribution
2. Ability to function over a wide range of sheet basis weights
3. Fine spatial resolution
4. Easily quantified results
5. Insensitivity to presence of additives, colorants, etc.
6. Fast, easy operation
7. Large sample size.

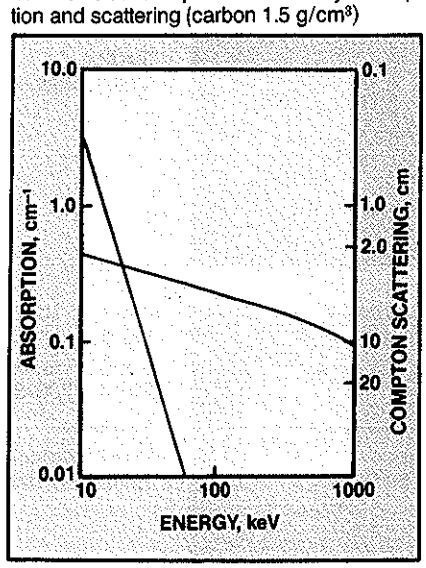
Soft x-ray imaging

X-ray energies can be grouped as shown in **Table I.** While x-rays are attenuated at all energy levels according to Eq. 1, the value of μ, the linear absorption coefficient, varies with energy and from material to material in a well-documented fashion.

I. X-ray energy levels

	Energy, keV	*Wavelength, A*
Ultrasoft	0.12–1.2	10–100
Soft	1.2–12.0	1.0–10
Hard	12–120	0.1–1.0
Ultrahard	>120	<0.1

1. The relative importance of x-ray absorption and scattering (carbon 1.5 g/cm³)



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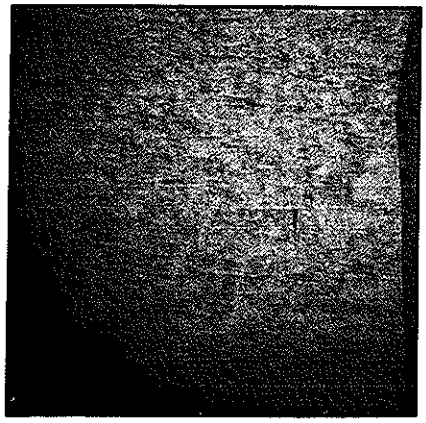
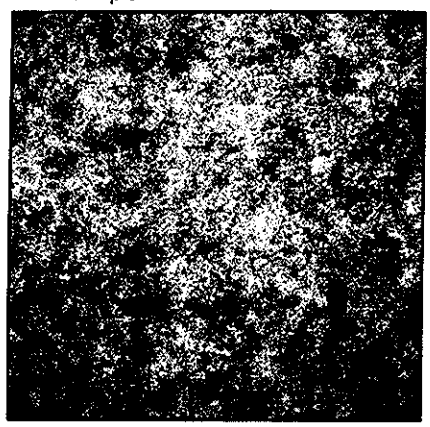
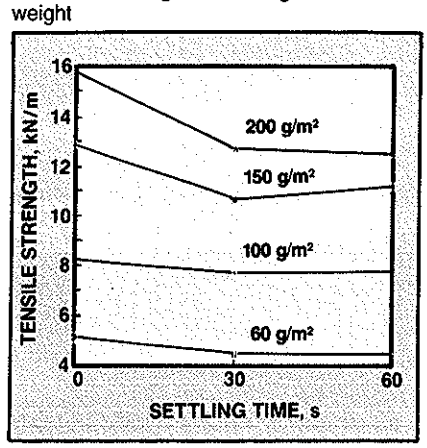
2. Soft x-ray image of a commercial tissue sample



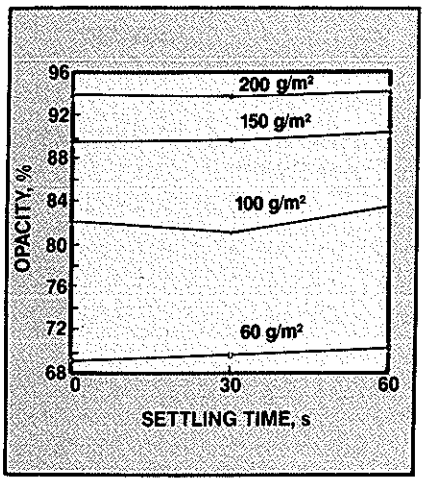
3. Soft x-ray image of a commercial linerboard sample



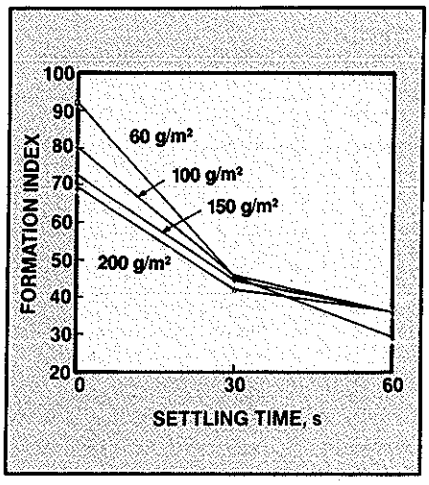
4. Tensile strength vs. settling time and basis weight



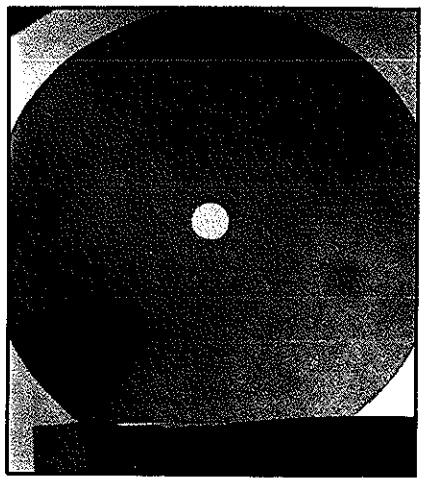
5. Opacity vs. settling time and basis weight



6. Thwing Formation Index vs. settling time and basis weight



7. Typical radiograph with calibration wedge (60/std)



$$I_{out} = I_{in}\, e^{-\mu l} \qquad (1)$$

where

I_{out} = x-ray intensity leaving the sample

I_{in} = incident x-ray intensity

μ = linear absorption coefficient, cm^{-1}

l = sample thickness, cm.

More importantly, several mechanisms contribute to total attenuation, as shown in Eq. 2.

$$(\mu/\rho)_T = (\mu/\rho)_A + (\mu/\rho)_S + (\mu/\rho)_{PP} \qquad (2)$$

where

ρ = density , g/cm^3

μ/ρ = mass absorption coefficient

T = total

A = ascribed to absorption

S = ascribed to scattering

PP = ascribed to pair production.

As energy increases, the relative importance of absorption, scattering, and pair production varies, as shown in **Fig. 1** for carbon. The usefulness of soft X-ray imaging for formation measurement is based on the fact that absorption dominates over scattering as the attenuation mechanism for carbonlike materials at energies below 10 keV.

This has two important implications. First, an image produced at such low energies must reflect the true mass. Second, the relatively low level of scattering should give fine spatial resolution.

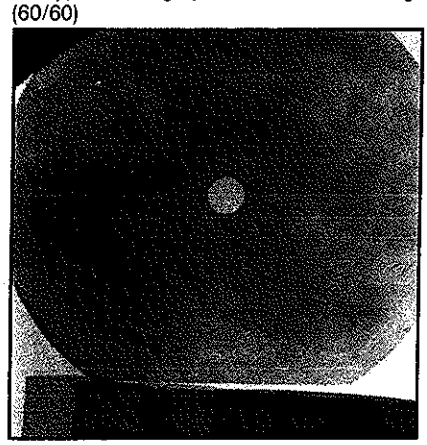
While the theoretical possibility for directly imaging true mass distribution with fine resolution is appealing, the first question must be a practical one. Can such low-energy imaging be conducted in reasonable times over a wide range of sheet basis weights? Initial experiments answered this question in the affirmative. **Figures 2 and 3** show radiographs of commercial tissue and linerboard samples. Conditions for these radiographs are listed in **Table II** (the energy units are kilovolts potential at the source).

Because the sheet samples are in intimate contact with the film, resolution is fine. Tests with fine metal filaments show spatial resolution to be in the range of 5–10 μm, with film

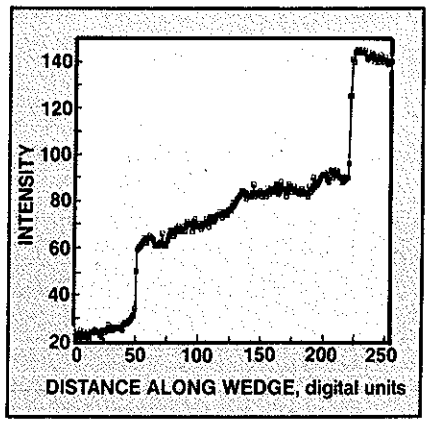
II. Initial trials

	Sample 1 (Fig. 2)	*Sample 2 (Fig. 3)*
Film	Kodak LP7	Kodak Ultratec
Energy, kVp	10	6
Developer	Kodak D-8	Kodak Ultratec
Developing time, min	3.0	1.5

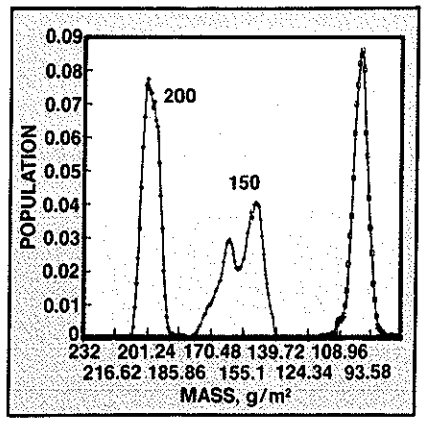
8. Typical radiograph with calibration wedge (60/60)



9. Spatial variation of intensity in wedge



10. Statistical variation of intensity in wedge



III. Handsheet data

Sample no.	Basis weight, g/m²	Settling trime, s	Tensile strength, kN/m	Opacity, %	Formation index
60/std	60	0	5.13	69.1	92.1
60/30	60	30	4.47	69.6	45.6
60/60	60	60	4.47	70.3	29.4
100/std	100	0	8.23	82.1	79.8
100/30	100	30	7.71	81.0	45.9
100/60	100	60	7.77	83.4	36.2
150/std	150	0	12.9	89.5	72.3
150/30	150	30	10.7	89.6	44.7
150/60	150	60	11.2	90.3	35.9
200/std	200	0	15.8	93.9	69.2
200/30	200	30	12.7	93.7	42.2
200/60	200	60	12.5	94.1	36.1

IV. Film processing conditions

Film	Kodak LP7
Exposure time, min	60
Energy, kVp	8
Developer	EKtaflow Type 1
Developing time, min	2

uniformity being the limiting factor. All radiographs produced in this study were taken with a commercially available cabinet x-ray system (Hewlett-Packard Faxitron Model 43855A).

Measurement of density distribution

To assess the utility of this technique for formation measurement, a number of handsheets were produced on a standard former. Sheets were produced at basis weights of 60, 100, 150, and 200 g/m². At each basis weight, sheet formation was varied by allowing the suspension to settle for times of 0 s (standard), 30 s, and 60 s prior to sheet production. These sheets were pressed, air dried, and analyzed for tensile strength, opacity, and formation (Thwing-Albert). These results are listed in **Table III** and are depicted in **Figs. 4–6.**

All samples were imaged using the cabinet x-ray system. Processing conditions are listed in **Table IV.** A step wedge of 50-g/m² mylar sheets served as a calibration for each shot. Typical images are shown in **Figs. 7 and 8.**

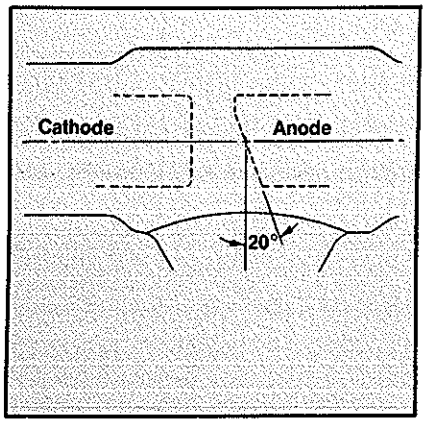
Figures 9 and 10 show image intensity for the calibration wedge. All radiographs were reverse printed to give positives for image analysis. Analysis was performed on a Tracor-Northern image analyzer. Figure 9 shows the spatial variation of intensity across part of a wedge, while Fig. 10 depicts the statistical distribution of intensity vs. basis weight. Intensity distributions have been normalized to give equal areas under the peaks corresponding to 100, 150, and 200 g/m² in Fig. 10.

The presence of a double peak at 150 g/m² indicates one of the fundamental problems with the technique. Due to the X-ray source "heel" effect shown in **Fig. 11**, there is a significant variation of x-ray field intensity within the cabinet, as shown in **Fig. 12.** While no attempt was made to account for this variation in the intensity data, its effect was minimized by studying relatively small samples at the maximum allowable source-to-film distance.

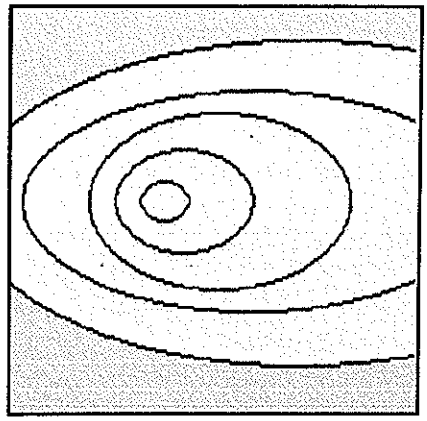
The raw intensity distributions for samples 150/std and 150/30 are shown in **Fig. 13.** Sample fields of 3.75× 3.75 cm were digitized with 512 × 512 resolution for spatial resolution of approximately 70 μm. Statistical data for these images and for a blank exposure of comparable average exposure are listed in **Table V.** Variability in average intensity for samples of the same basis weight can be traced to exposure and manual processing variability, both of which can be eliminated.

The intensity histograms were normalized, translated to give equal average values, and converted to basis weight histograms using the slope of the calibration correlation shown in **Fig. 14.** The resulting histograms and statistics are shown in **Fig. 15** and **Table V**, respectively.

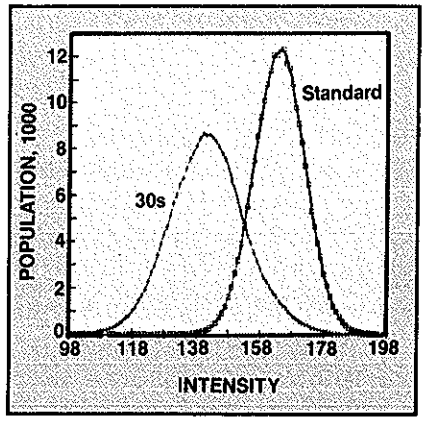
11. X-ray source heel effect



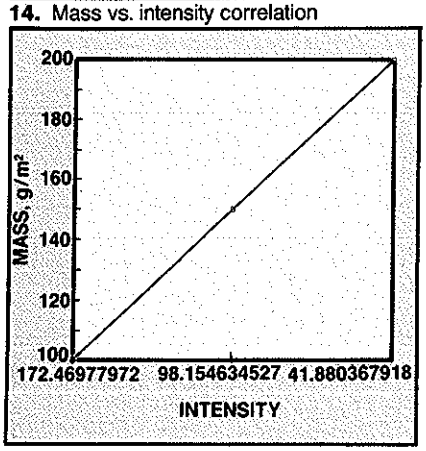
12. Spatial variation of x-ray field intensity



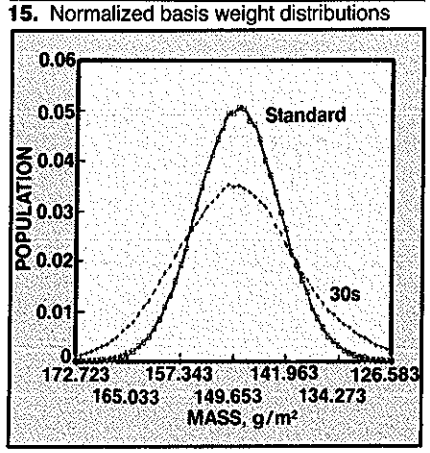
13. Raw intensity distributions



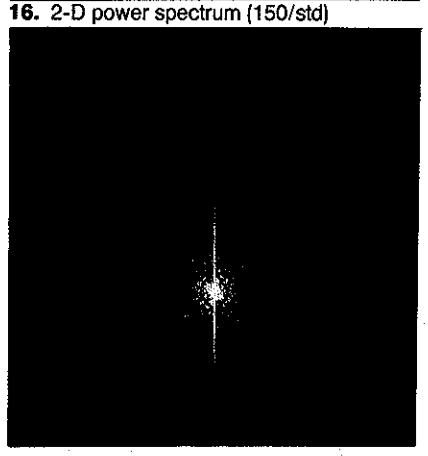
14. Mass vs. intensity correlation



15. Normalized basis weight distributions



16. 2-D power spectrum (150/std)



Even with the problems of spatial variation of the x-ray field and sample-to-sample processing variations, soft x-ray imaging is capable of quantifying mass distribution when coupled with digital image processing. Spatial resolution is limited not by the imaging technique but by digital processing resolution.

V. Raw intensity data and normalized basis weight distributions

		Raw data		*Normalized*	
	Blank	*150/std*	*150/30*	*150/std*	*150/30*
Average	152.0	164.0	141.6	148.9	150.1
Variance	17.9	65.0	144.1	38.5	85.2
Std. dev.	4.2	8.1	12.0	6.2	12.3

Floc size distribution

While the statistical distribution of sheet density is important, at least as important is some measure of its scale of spatial variation as it relates to floc size distribution. To assess spatial periodicity of density distribution, the samples were analyzed via two-dimensional Fast Fourier Transforms (FFT). The 2-D power spectra for samples 150/std and 150/30 are shown in **Figs. 16 and 17.** The broad power spectrum observed in Fig. 16 is indicative of the fine structure (high frequency variation) dominating the well-formed sample, while the more compact spectrum of Fig. 17 can be attributed to the large-scale variations (floc structure) present in the less even sample.

While it is difficult to correct for the spatial variations of the x-ray field in physical space, it is relatively simple to account for them in Fourier space because they superimpose long wavelength variations on the image. **Figures 18 and 19** were obtained by subtracting the power spectrum of the blank exposure room from those of the two sheet samples. Note that Fig. 19 has been magnified 3X now. Again, the broader spectrum is indicative of finer structure in the sample. Some measure of this breadth, such as half height width, can be used to "put a number on" this indicator of floc size distribution.

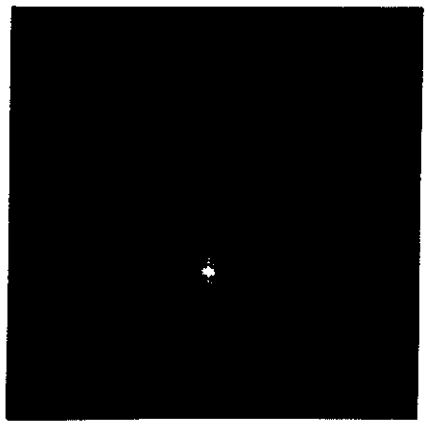
Summary of the formation tests

The main objective of these tests was to determine if soft x-ray imaging can serve as a practical tool to assess sheet density and flow size distribution. Difficulties were encountered which can be traced to the variability in x-ray field intensity and processing. The efforts of Yuhara[1] and Hasuike[2] have shown that x-ray field intensity variations can be corrected for by

[1]Yuhara, T., Hasuike, M., and Murakami, K., Japan TAPPI 40(1): 85(1986).

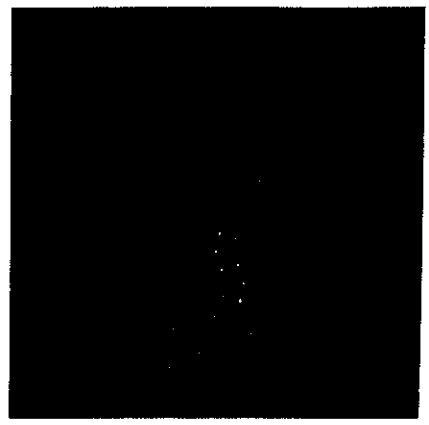
[2]Hasuike, M., private communication.

17. 2-D power spectrum (150/30)

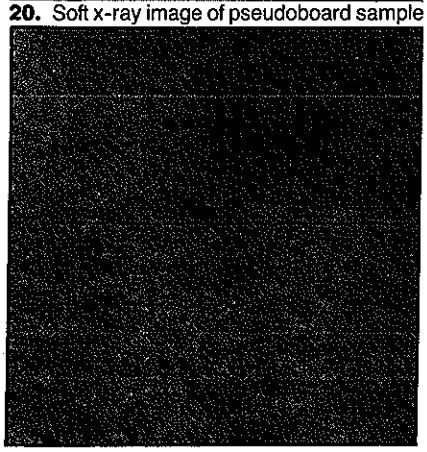


18. 2-D power spectrum (150/std minus blank)

19. 2-D power spectrum [(150/30) minus blank (at 3X)]



20. Soft x-ray image of pseudoboard sample



21. Flash x-ray image of pseudoboard

digitizing a blank image and using it in a pixel-by-pixel correction scheme.

While state-of-the-art image analysis can be employed to obtain quantitative data on both density and floc size distribution, the question is, are these tools available to those most interested in measuring sheet quality? The answer may be found in optical image processing. Most of the computation performed digitally in this study can be executed via relatively inexpensive optical techniques at much greater speed and finer resolution.

Other features of soft x-ray imaging

Any optically based formation tester must be sensitive to the presence of fine particles—additives and colorants in particular. Since these materials are usually present at low levels on a mass basis, a true density distribution measurement such as soft x-ray is not significantly disturbed.

Low-energy x-ray imaging can be used to monitor contaminant levels. **Figure 20** identifies the particles contaminating a piece of pseudoboard, and the resulting image can be easily processed digitally to obtain particle size distribution data.

While all images produced in this study employed a cabinet x-ray system and exposure times on the order of seconds to minutes, it is possible to obtain images at speeds which make on-line measurements at least feasible. **Figure 21** is a flash x-ray radiograph of the pseudoboard sample. Some resolution and contrast are lost relative to the cabinet pictures as the effective energy level is approximately 20 keV. However, the flash image exposure time was 70 ns.

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