TOPOGRAPHY OF GLOSS DETAILED WITH THE SCANNING ELECTRON MICROSCOPE

Lois A. Settlemeyer, *

Abstract: The scanning electron microscope (SEM) has long been recognized as a tool to give a visual image of a surface. Because of the SEM's wide magnification range both macro and micro structure can be recorded. A method which uses the SEM to record coated paper topography and provide an understanding of surface structure is described in this paper. The method is especially useful when the typical gloss measurement tools do not agree with visual perception. High gloss, depth of gloss, or icy gloss are better visualized with this new procedure. The new method differs from normal SEM operation in that the sample angle is set to be the complement of the gloss measurement tool angle.

This paper will first review how macro and micro structure effects gloss, how typical gloss measurement tools can be misleading, and how the human eye differs in its perception from these tools. Photomicrographs will highlight the advantages the new SEM method offers. How topography relates to gloss will be documented on fine paper vs cast coated paper, machine vs cross machine direction, the development of gloss via calendering , and ink gloss reduction due to heat.

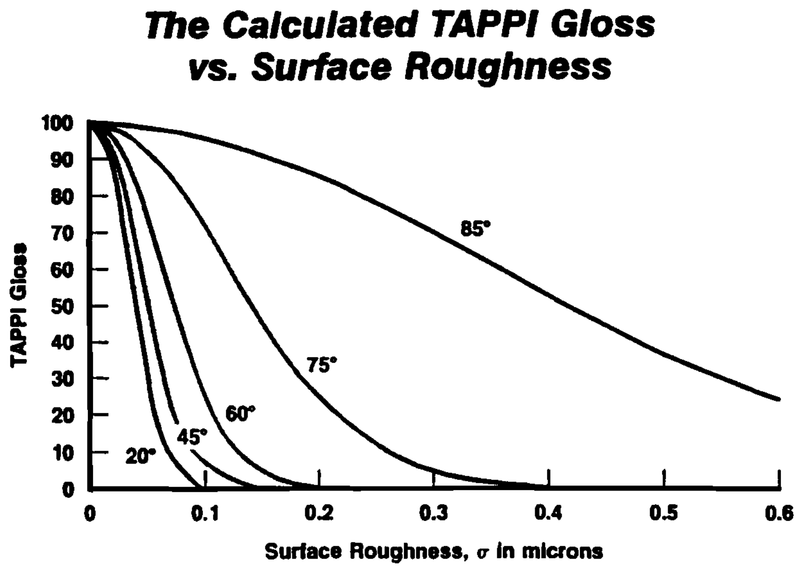
I. Application of Gloss Measurement Tools to Surface Smoothness

Surface imperfections greater than 0.03 micron interfere with gloss. Large paper fibers or machine flaws are 10-12 microns and contribute to surface imperfections that are described as macroroughness. Pigment particles (~1 micron) and binders (~ 0.15 micron) contribute microroughness to the sheet. Due to the large size range influencing the paper surface, and the single angle of light reflection measured, typical gloss measurement tools measure only one

*Development Leader, Latex Coatings and Binders TS&D,
2040 W. H. Dow Center, Dow Chemical Company, Midland, Mi. 48674

level of roughness, either micro or macro. This contributes to the discrepancy between visual and measured gloss.

Lee, et.al. presented the calculated TAPPI Gloss as a function of surface roughness (Graph I) . This type of numerical record assists in understanding surface structure and gloss. However, it is difficult for most individuals to correlate numerical values with topography. A visualization of the micro and macro surface roughness is needed.



Graph I. The Calculated TAPPI Gloss vs. Surface Roughness

II. Introduction To Gloss Measurements

Gloss is one of the most critical properties of the printed page. Ink gloss and sheet gloss are both equated with aesthetics and quality. Technically defined, gloss is the geometrically selective reflectance of a surface. To the human eye, however, gloss depends upon the light source, the object, and the observer. The observer translates the image into a perception or a psychological reaction. Quantifying the psychological perception by giving a numerical value via a gloss measurement tool, can be misleading.

II. A. Glossmeters

Glossmeters are photoelectric instruments which indicate in percent the amount of light specularly reflected from a surface relative to a standard. In the paper industry the spectral angle is typically 20°, 60°, or 75°. A polished black glass with a known refractive index reflects 100% light in the specular direction and is the calibration standard. TAPPI Official Test Methods T480 and T653 specify the geometric, photometric and spectral requirements of the standard glossmeter. Thus, the light source, the angle of incidence and the angle of viewing are selected variables. Twenty degree gloss is typically used for high gloss specimens, but a large grazing angle of about 75° is better for low gloss samples in order to obtain sufficient reflectance to differentiate among the samples.

For this paper, a Technidyne Model T480A has been used for all gloss measurements.

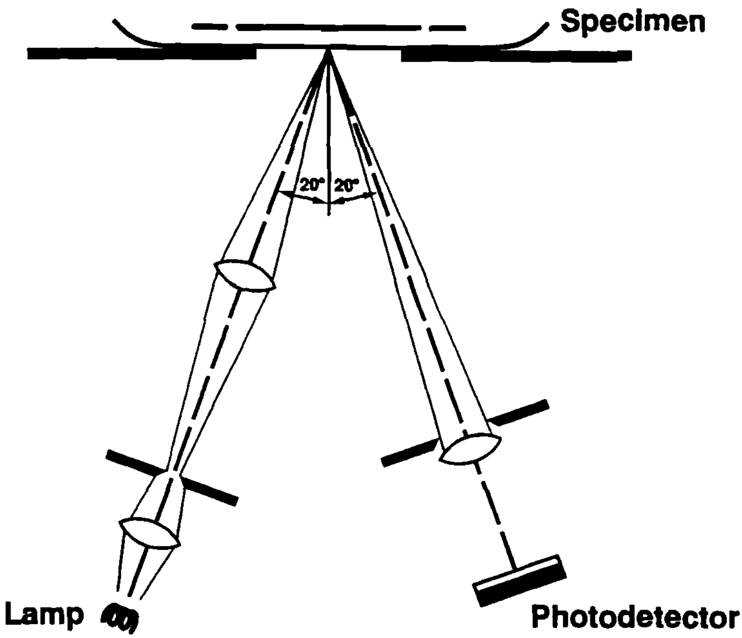


Illustration I. Glossmeter Optical System

II.B. Goniophotometers

A goniophotometer measures the distribution of light reflected from a surface over a range of collector angles. Both the light source and the collecting photometer position can be varied independently. Collected data requires extensive interpretation: peak height, the half-width at half the peak height, and the area under the curve. Due to the complexity of potential angles and interpretation most goniophotometer measurements are made under a specific set of conditions.

The goniophotometer readings presented in this paper were recorded with a HunterLab Dorigon II. This is a hand-held or bench-top unit using a broad band wavelength light source centered in the visible region and 30° from the perpendicular of the sample. The specular reflectance and the diffuse angle is measured with a series of 5 detectors, one at the specular angle, two at 0.3° either side of the specular angle detector which measure "distinctness of image" (DOI), and two detectors at 2° either side of the specular angle which measure "Haze". DOI and the HAZE data are ratios of the reflected light of the variable to the spectral light.

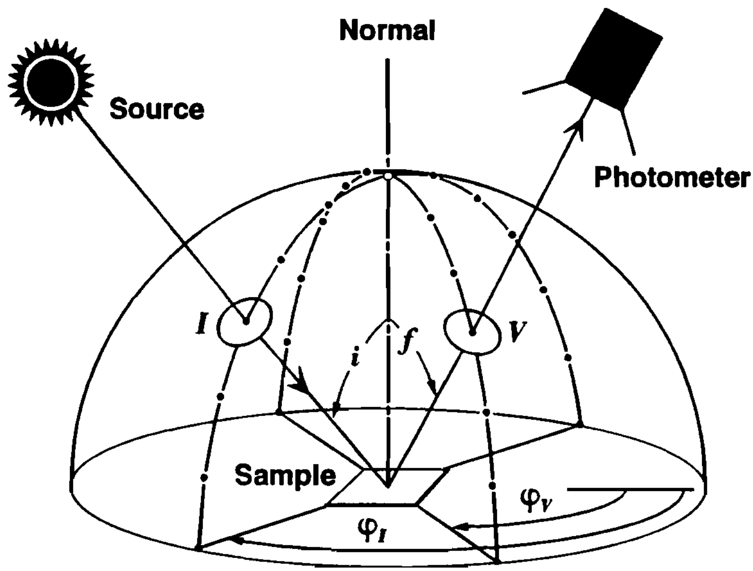


Illustration II. Schematic Diagram of Goniophotometer

II. C. Profilometer

The profilometer is an instrument originally designed to measure the surface smoothness of polished metals. The instrument measures the profile of a surface by moving a stylus over the surface and recording the calibrated amplified motion of the stylus. The size and type of stylus are critical to the level of macro/micro roughness measured. This instrument is sensitive to both machine and cross machine direction due to the macro roughness caused by the fibers.

II. D. The Human Eye

The function of the human eye is very different than that of gloss measurement tools. Light enters the eye through the pupil and is focused on the retina. This light is from all portions of the field of view, and size, shape, depth, shadow, and distance, are all involved in perceiving the image. Likewise, before any perception can occur, the light striking the retina must be absorbed and converted into a physiological message that is transmitted to the brain. In the brain the message is interpreted and perception or a mental image is formed. (Overheim, 1982).

In general the light reaching our eyes from an object is not uniform. For example, hills are brighter than valleys; glossy objects are brighter than mattes. It is the presence or absence of these highlights that alerts the mind to the topography or glossy nature of a surface.

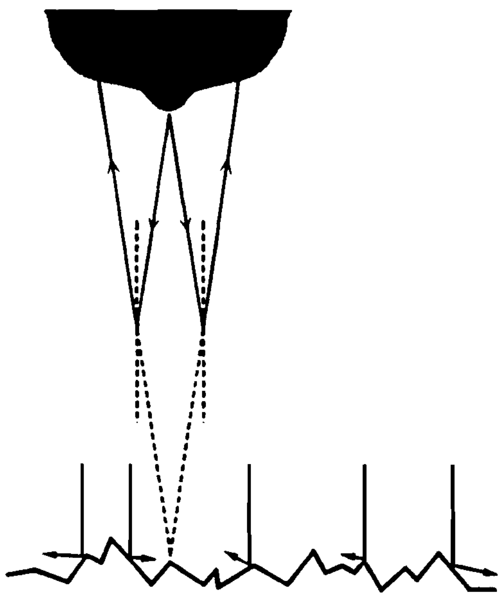


Illustration III. Viewing a Reflective Surface

III. Application of the SEM to gloss

The scanning electron microscope (SEM) is a high resolution instrument that can record surface topography. An electron beam is used to illuminate the sample. The beam is focused with the use of electromagnetic lenses and scans across the sample surface. Low energy secondary electrons are emitted from the sample surface, collected and transmitted to a cathode ray tube to form an image. The image, or surface topography of the sample, is recorded on a photomicrograph. Contrast in photomicrographs is dependent upon the number of secondary and backscattered electrons emitted from the sample surface. Peaks, valleys, density, and elemental differences all contribute to image contrast.

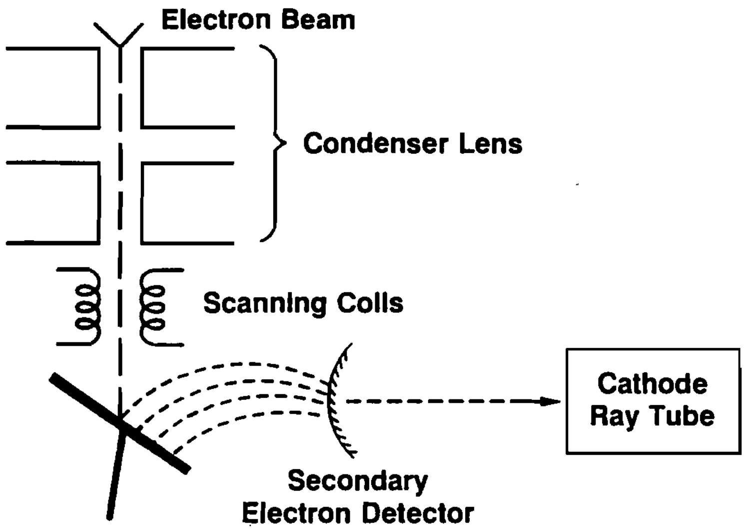


Illustration IV. The Scanning Electron Microscope

The scanning electron microscope has long been used to observe both microroughness and macroroughness via a high resolution visual definition. Since the angle of the electron beam is stationary, the angle in which the beam strikes the surface of the sample is dependent upon the tilt of the sample. In most instruments, sample angle can be adjusted from 0 to 90 degrees, but for ease of operation and improved resolution, the SEM is typically operated at a sample angle of 30° to 45°. These conditions are best for recording porosity and micro detail.

An Amray 1810 SEM was used with the angle of the sample being adjusted to be equal to the glossmeter incident light angle for the work recorded in this paper.

Illustration V diagrams the key to the proposed new method. Whereas gloss instruments measure the specular angle of the light source with relation to a stationary sample, in the SEM the electron beam is stationary and the sample angle is variable. Photomicrographs recorded at a sample angle of 15° show the topography the glossmeter measures at 75°, and photomicrographs recorded at a sample angle of 70° show the topography the glossmeter measures at 20°. Just as the 20° glazing angle is useful in the gloss meter for measuring high gloss samples, the 70° angle in the SEM is especially useful for recording topography which is characterized as depth of gloss or icy gloss.

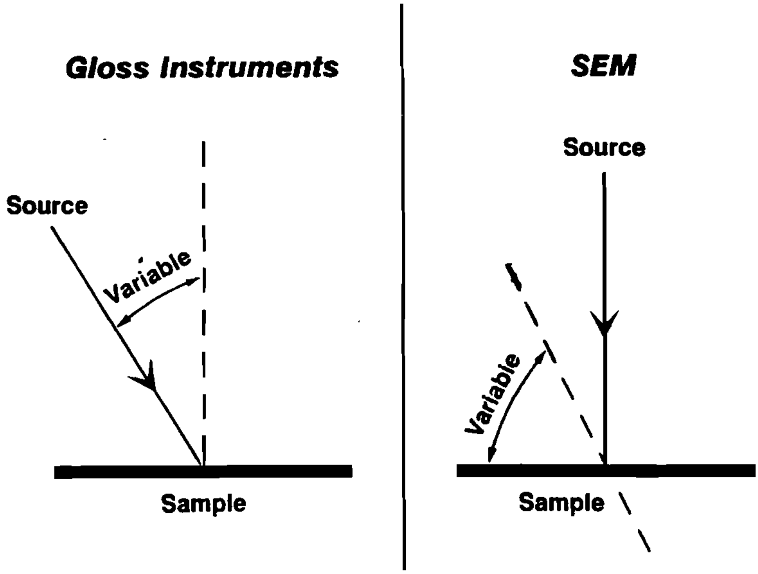


Illustration V. Measured Angle via Gloss Instrument vs. SEM

IV. Observations and Data Evaluation

IV. A. Fine Paper Topography Using the SEM

A #1 grade sheet of fine paper had a measured 75° sheet gloss of 83 ± 0.5, a distinctness of image (DOI) of 6.5 ±1.5 and a haze of 49 ±4. (Table I) The series of photomicrographs records SEM sample angle differences over a range of magnifications. The photomicrographs are connected in serial order to produce a greater surface area for comparison. The contrast differences at the low sample angles are due more to density and elemental change rather than topography. The high angle, (70°), series does not show fine detail, density or elemental differences. It does produce visually a surface topography which will guide understanding of the glossmeter, goniophotometer, and profilometer data. (Photomicrograph Figures I, II, and III)

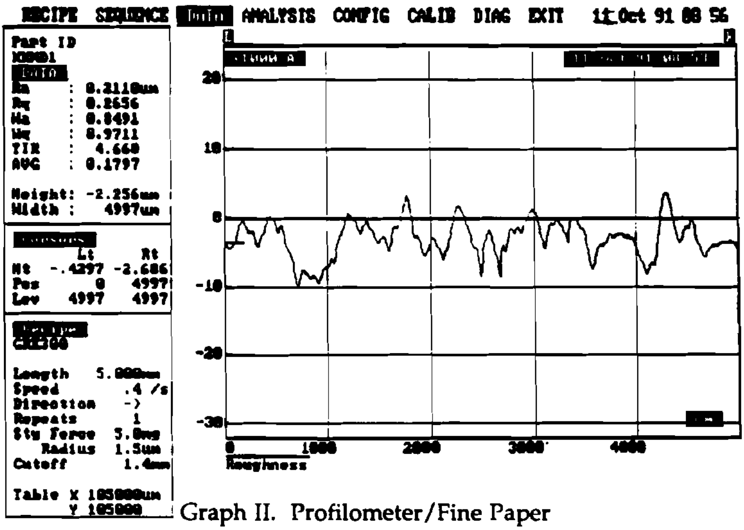
Figure	Sample ID	Sheet Gloss 75°	60°	30°	20°	DOI	Haze
1, 2, 3	#1 Grade	83 ± .5		4.5 ± .3		6.5 ± 1.5	49 ± 4
4, 5, 6	Cast Coated	83 ± .6		11 ± .7		30 ± 3.5	3.6 ± .4
7, 8, 9	#1 Grade (CMD)	83 ± .5		4.6 ± .3		4.0 ± .5	20 ± 5
10, 11, 12, 13, 14	0 Nip	29 ± .5			4.3 ± 0		
	1 Nip	67 ± .8			11.1 ± .6		
	2 Nip	73 ± 1.4			15.4 ± .7		
	3 Nip	76 ± .8			16.1 ± .7		
15, 16	Ink Gloss						
	Low Temp.	76			18.5 ± .7		
	High Temp.	71			13.7 ± .7		

± = 1α

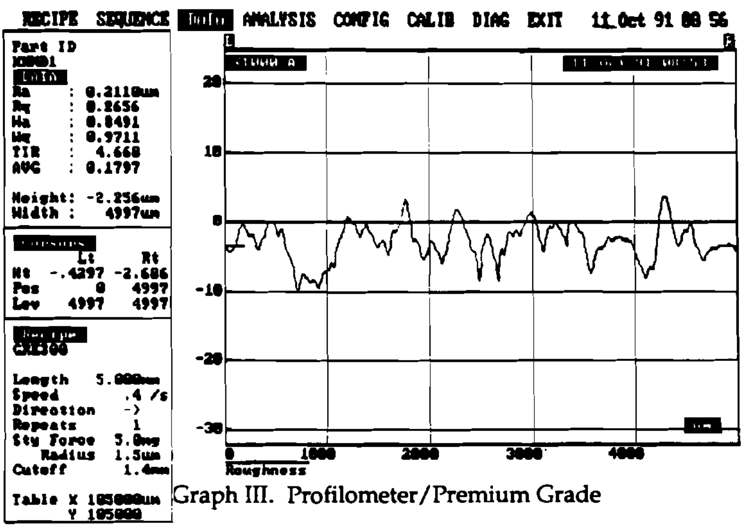
Table I. Glossmeter and Goniophotometer Values

IV B. Fine Paper Evaluation vs. Premium Grade Sheet

The second series of photomicrographs compare a number one grade fine paper with a cast coated premium grade. The 75° sheet gloss is measured as 83 for both sheets. The goinophotometer records the DOI and the HAZE as being much higher for the #1 sheet than for the premium grade sheet. In contrast to these values, the human eye would see the premium grade sheet as having superior gloss. The profilometer records the surface variations, graph II and III, as being insignificant.



Graph II. Profilometer/Fine Paper



Graph III. Profilometer/Premium Grade

Comparing photomicrographs, it is obvious that the #1 sheet does not have the level of micro smoothness displayed on the premium sheet. The equal gloss reading on the cast coated sheet with the #1 sheet is due to the machine flaws and not the coating controlled micro surface structure.
(Compare photomicrographs IV, V, VI with I, II, and III)

IV C. Fine Paper in Machine and Cross Machine Direction

Using the same #1 grade sheet as above, gloss and topography were characterized and compared in the machine and cross machine direction. Visually the human eye would see a difference in gloss, however both the 75° and the 20° gloss measurements did not measure any difference. The goniophotometer did measure a gloss difference in the distinctness of image and haze. (Table I.)

Photomicrographs record the topographical differences for the visual gloss reduction in the cross machine direction.
(Photomicrographs VII).

IV D. Calendering Gloss Development

The primary reasons for coating and calendering paper are to reduce the surface roughness, improve gloss, and improve print quality. In gloss development studies, the gloss of a sheet is measured prior to calendering and following the first, second and third calender nips.

The low SEM angle photomicroscopy, 15° and 30°,shows density differences which the high angle, 70°, does not show. For instance, at 15° each increasing nip shows the increased density of the pigment over the large surface fibers. High density compaction is generally identified as calender blackening. At the intermediate SEM angle, 30° , a decrease in porosity is evident as the number of nips increases. However, it is the 70° angle that shows the topography which relates to gloss. The leveling of the macro roughness with each increasing nip improves the gloss (Figure XI), but the micro roughness associated with the filler distribution (Figure XII) and placement remains unchanged. (Compare photomicrographs VIII, IX, X, XI, and XII,

IV E. Ink Gloss

Ink gloss can be reduced during ink set in web offset drying ovens. This loss is usually the result of elevated drying temperatures. Photomicrographs XIII and XIV compare the surface of two black heat-set inks of equal density. One sample was dried at an oven air temperature 100°F lower than the other. Ink gloss measurements recorded a five point difference, with the higher air temperature having the lower gloss. Fiber roughening or fiber puffing was the anticipated reason for the gloss reduction. (Compare photomicrographs XIII and XIV).

The photomicrographs show a micro roughness on the high temperature ink surface which, due to its size, probably does not relate to fiber roughening but rather to ink penetration and roughening.

V. Summary And Conclusion

The scanning electron microscope can be used as a tool to visualize the topography which influences gloss. The new method records macro and micro structure contributing to topography and provides an understanding of surface structure when the typical gloss measurement tools do not agree with visual perception. The key to enhanced definition is the sample angle: an adjustment of the sample in the electron beam to equal the specular angle of the gloss measurement tool. Just as the gloss meter at a 20° glazing angle is especially useful for measuring high gloss samples, the 70° angle in the SEM is especially useful for recording topography.

This method has been applied to visualize fine paper gloss, premium paper quality, machine direction and cross machine variations, calendering gloss development and loss of ink gloss due to drying conditions. Visualization of the topography which contributes to gloss should be a valuable long-term addition to a paper characterization data base as it improves our understanding of how the human eye integrates micro and macro roughness into an image called gloss.

Figure I #1 FINE PAPER SHEET, 15° SEM Angle

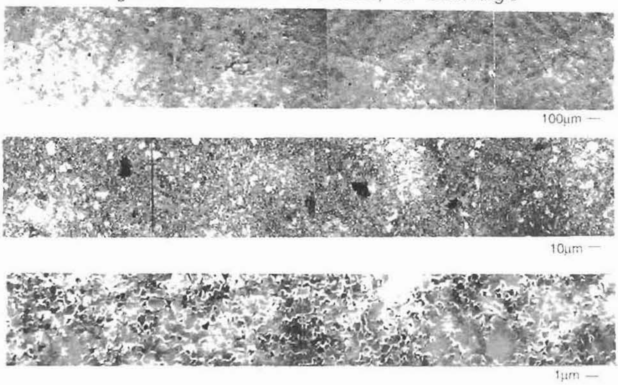


Figure II #1 FINE PAPER SHEET, 30° SEM Angle

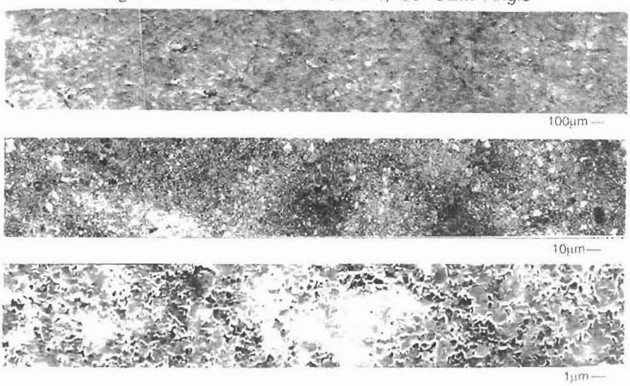


Figure III #1 FINE PAPER SHEET, 70° SEM Angle

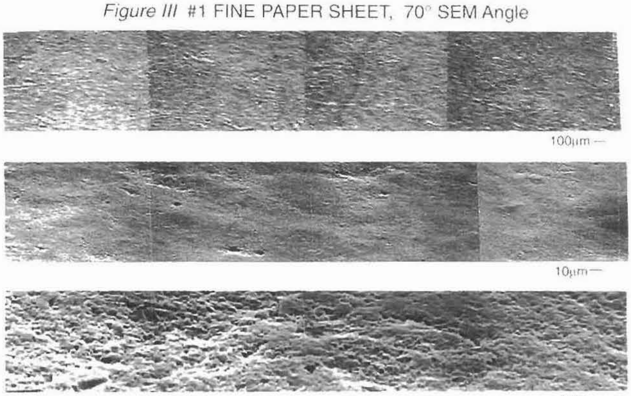


Figure I, II, III, Fine Paper Sheet

Figure IV CAST COATED, 15° SEM Angle

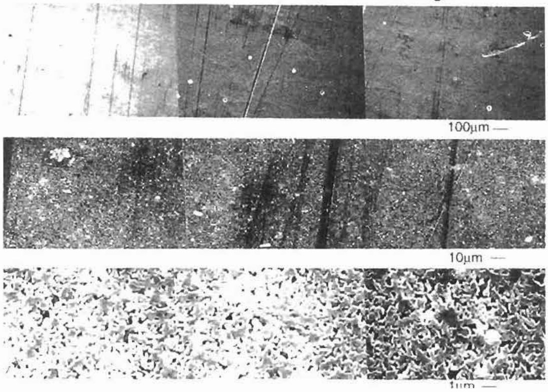


Figure V CAST COATED, 30°SEM Angle

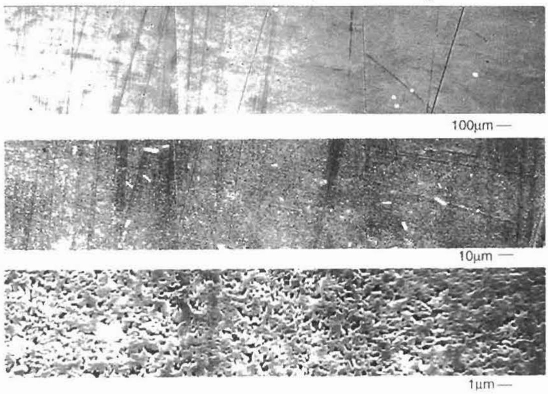


Figure VI CAST COATED, 70° SEM Angle

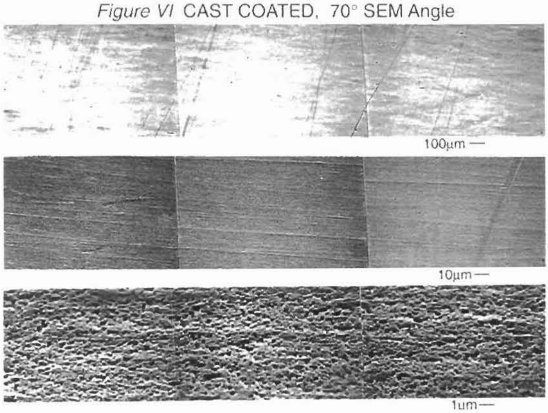


Figure IV, V, and VI. Cast Coated Sheet

Figure VII MACHINE vs CROSS MACHINE DIRECTION
#1 Fine Paper Sheet, 70° SEM Angle

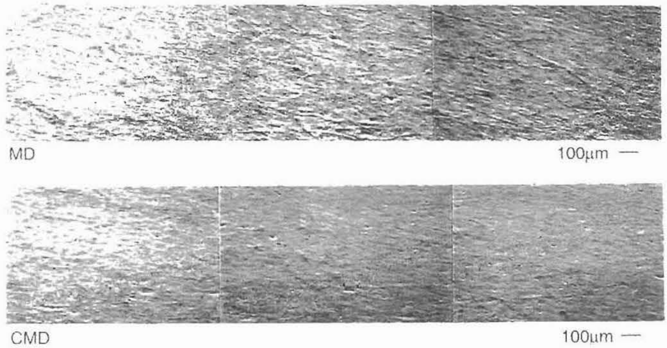


Figure VIII MACHINE vs CROSS MACHINE DIRECTION
#1 Fine Paper Sheet, 70° SEM Angle

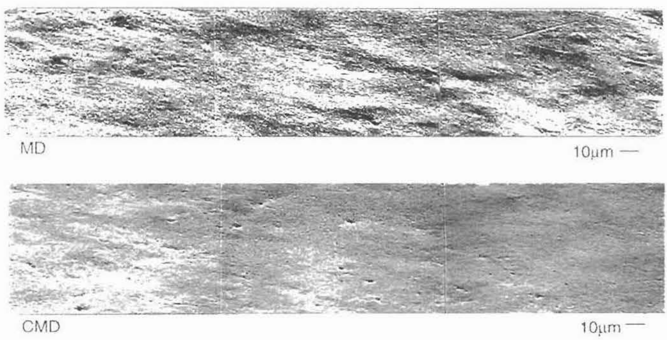


Figure IX MACHINE vs CROSS MACHINE DIRECTION
#1 Fine Paper Sheet, 70° SEM Angle

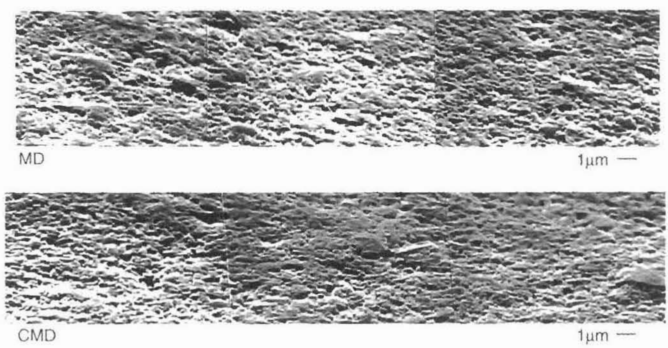


Figure VII, VIII, and IX. Machine vs Cross Machine Direction

Figure X GLOSS DEVELOPMENT, 15° SEM Angle

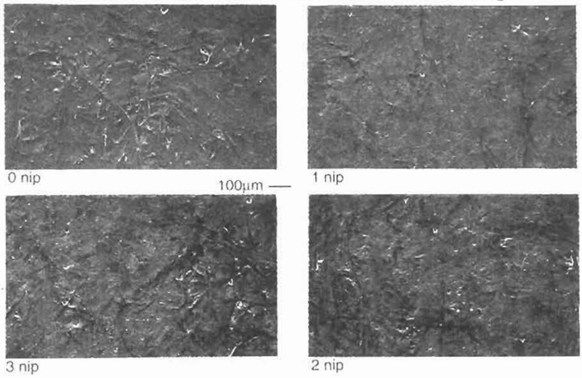


Figure XI GLOSS DEVELOPMENT, 15° SEM Angle

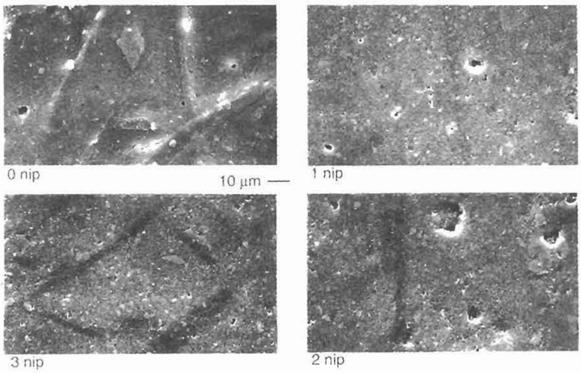


Figure XII GLOSS DEVELOPMENT, 30° SEM Angle

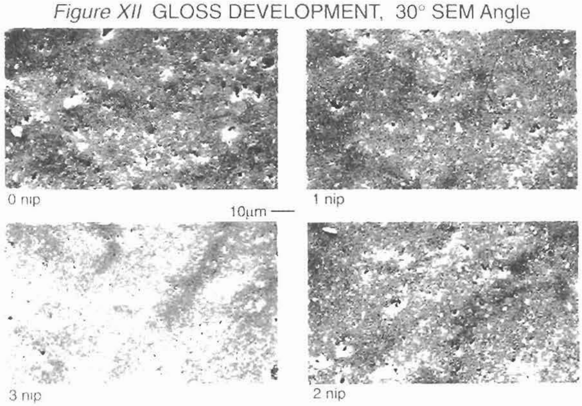


Figure X, XI, XII. Gloss Development

Figure XIII GLOSS DEVELOPMENT, 70° SEM Angle

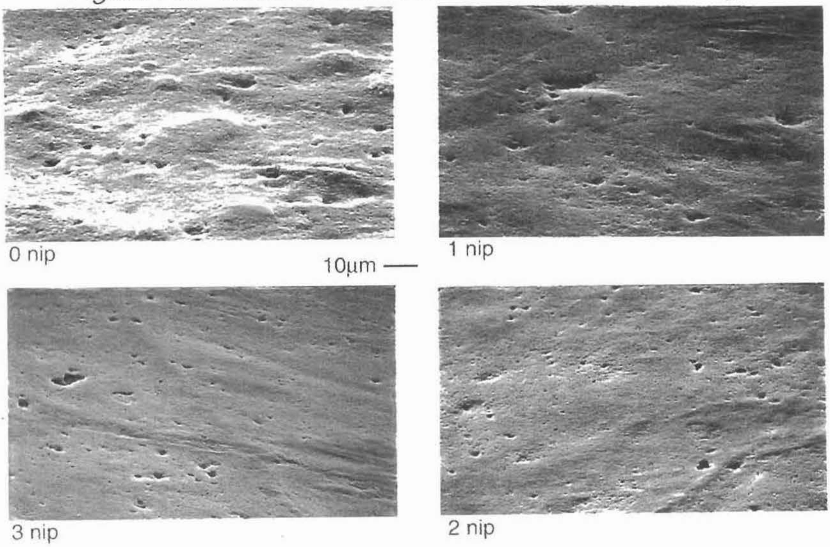


Figure XIV GLOSS DEVELOPMENT, 70° SEM Angle

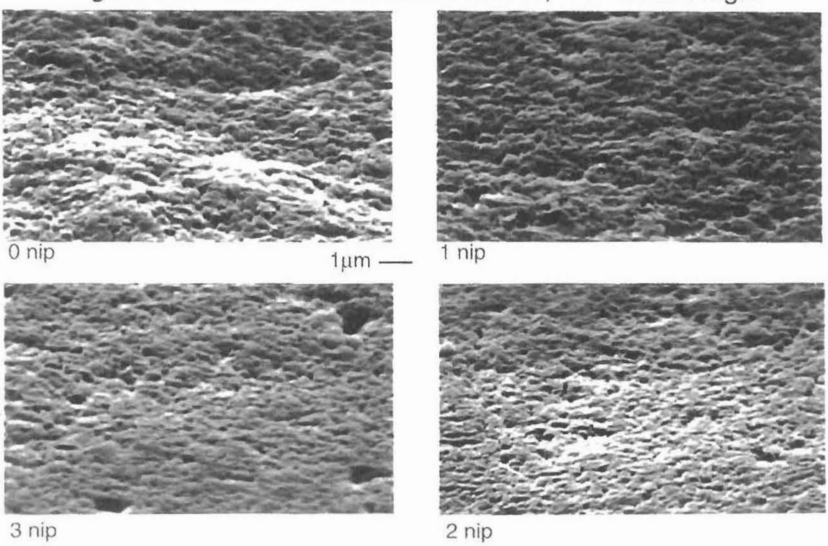


Figure XIII and XIV. Gloss Development

Figure XV INK GLOSS, 30° SEM Angle

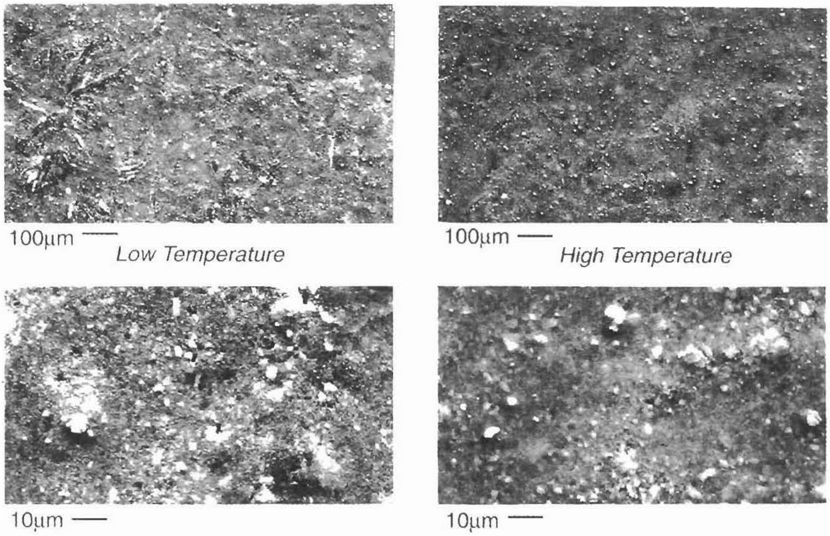


Figure XVI INK GLOSS, 70° SEM Angle

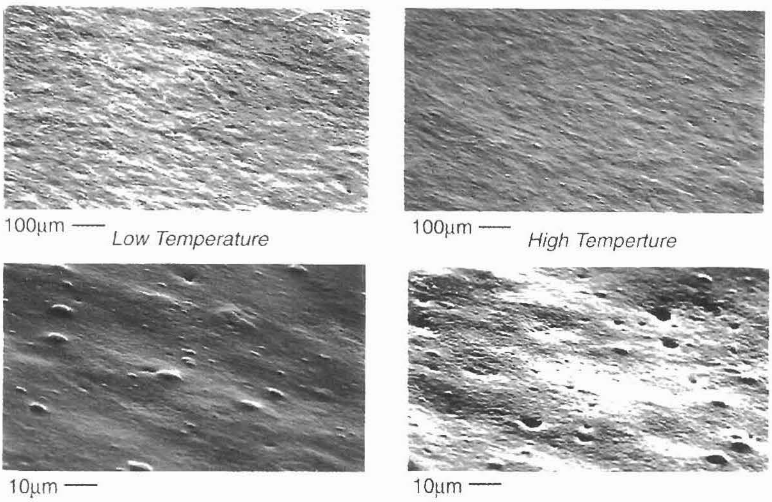


Figure XV and XVI. Ink Gloss

VI. Literature Cited

Funk, E.T., Loucopoulos, L., and Vogel, T.C.
1988. "Using the Scanning Electron Microscope to Augment Standard Paper Testing Methods" (Gravure Association of America.

Gate, L.
1973. "The Gloss of Coated Paper," TAPPI Vol. 56 No. 3, 61-65 pp.

Lavelle, J.S.
1982. "Gloss: Theory and Its Application to Printed Ink Films," National Printing Ink Research Institute, Lehigh University.

Lee, D.I., and Hendershot, R.E.
1986. "Development of Low Glossing Paper Coating Latexes: Theories and Concepts," TAPPI Coating Conference Proceedings.

Lipshitz, H., Gridger, M. and Derman, G.
1990. "On the Relationships Between Topography and Gloss," TAPPI Journal, October, 237 pp.

Overheim, R.D. and Wagner, D.L.
1982, "Light and Color," (John Wiley and Sons, Inc.), Copyright 1982, 85 pp.