Polarized light reflectometry for studies of paper coating structure Part II. Application to coating structure, gloss and porosity

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ABSTRACT: Polarized light reflectometry is a useful noncontact method for obtaining information about paper coating structure, roughness, and optical properties. Basic data obtained from the reflectometer include refractive index, microroughness (surface roughness smaller than the wavelength of light), macroroughness (surface roughness greater than the wavelength of light), and various gloss and reflectance parameters. We used polarized light reflectometry and other complementary techniques to analyze a wide range of coated papers from pilot and mill trials. That allowed us to examine how the effective refractive index relates to more conventional measures of coating porosity. We found, for example, that refractive index correlates well with pore diameter as determined by mercury intrusion porosimetry. We discuss the measurements in terms of coating structure and show how the combination of data obtained by reflectometry can provide useful insights into coating structure, permit distinction between coating pigment and base sheet roughness features, and help quantify physical origins of sheet gloss.

Application: Polarized light reflectometry is a technique for measuring surface microroughness, macroroughness, and refractive index, which can help explain gloss and surface porosity properties.

Paper-coating structure determines most of the key properties of a finished sheet, including visual appearance and printability. Factors such as roughness, porosity, composition, homogeneity, isotropy, and differences between coating surface and bulk are all important.

Many studies of coating structure have been published and a wide variety of analytical techniques have been brought to bear on the problem. Examples include optical and electron microscopy [1-3], nuclear magnetic resonance (NMR) imaging [4], secondary ion mass spectrometry (SIMS) and X-ray photoelectron spectroscopy (XPS) [5], mercury porosimetry [6-8], profilometry [9, 10], atomic force microscopy (AFM) [11], X-ray diffraction (XRD) and fourier transform infrared spectrometry (FTIR) [12, 13], and gloss goniophotometry [14-17]. Some of these techniques are destructive; most are also slow and rather specialized, yielding detailed information on a specific aspect of the coating structure. Moreover, some techniques, such as mercury porosimetry and XRD, do not readily distinguish between coating and base sheet effects.

Previous work on gloss goniophotometry of coated paper has suggested the potential of polarized light reflectometry for structural studies, but wider application has been limited by the nature of the apparatus: mechanical goniometers tend to be slow, expensive, and rather specialized in use. In Paper I [18] we describe a recently developed imaging reflectometer that uses solid state imaging technology rather than a mechanical goniometer to measure the intensity and angular distribution of forward scattered light. This approach eliminates moving parts and makes reflectometry more practical for industrial use. A single measurement provides a range of data, including effective refractive index (RI; a measure of surface composition and porosity), macroroughness (roughness greater than the wavelength of light), and microroughness (roughness less than the wavelength of light).

In this paper, we present results obtained from analysis of a variety of commercial and pilot coated papers. In particular, we show how optical reflectance measurements can reveal information about surface porosity, composition, and structure.

POLARIZED LIGHT REFLECTOMETRY

We described the technique of polarized light reflectometry in some detail in Paper I [18] and summarize the key points here for completeness.

A collimated beam of light is directed at the surface of a coated paper at a high angle of incidence and forward scattered about the specular angle. The angular distribution of the scattered light is determined by the roughness of the coated surface. Our model uses a two-scale roughness comprising a macroroughness component and a microroughness component. For the macroroughness component, the surface slope or facet distribution is largely determined by the base sheet surface and coverage of any coating. For the microroughness component, the roughness features are typically less than a few hundred nanometers in amplitude and are characteristic of the coating pigment and pigment packing. The intensity of the scattered light is also controlled by the effective refractive index of the surface, which is related to surface composition (meaning the net composition of the surface controlled by properties such as pigment type, pigment packing characteristics, coating color formulation, and surface porosity). If the paper is printed, then the effective refractive index is largely determined by the surface ink composition.

The reflectance of the coated or printed paper may be described (to first order) by the following model for the intensity I of light reflected at some direction defined by (θ_r, ϕ_r) into a solid angle $d\omega_r$, for an incident beam of intensity I_0 (Paper I) [18]:

$$I(\theta_r,\phi_r)\ d\omega_r = I_0 R(\theta,n) M(\theta,\lambda,\sigma) F(\theta_f,\phi_f)\ d\omega_f + scatte \quad (1)$$

The angles (θ_f, ϕ_f) represent the orientation of a surface facet such that its local specular reflection points towards (θ_r, ϕ_r) and $F(\theta_f, \phi_f)$ represents the normalized fraction of facets at this orientation. The angle θ is the *local* angle of incidence at the surface for the given facet and $R(\theta,n)$ the Fresnel factor according to the polarization state of the light, determined by geometry and refractive index (n). The equations relating the various angles are determined entirely by the geometry and the angle of incidence of the light relative to the nominal (mean) surface. The factor $M(\theta,\lambda,\sigma)$ represents the intensity loss due to microroughness (σ), which depends on wavelength (λ) and geometry. For most surfaces, scattering also may be significant and some light multiply scattered within the bulk of the coating may be reflected into the forward detector angles.

By appropriate use of polarized light at two or more wavelengths and by measurements of the angular distribution of forward scattered light the effective refractive index n, the microroughness σ, and the surface slope distribution F may be determined (largely) independently of each other.

PAPERS STUDIED

We measured a wide range of coated papers as part of this study. Many of the samples were characterized in detail as part of earlier studies [8]. The coatings contained a number of mineral types, including platey, blocky, fine, and coarse kaolins; precipitated calcium carbonates (PCCs); and ground calcium carbonates (GCCs). Coatings with each mineral sample were formulated with between 9 and 12 wt% (expressed on pigment) of a carboxylated styrene-butadiene latex binder (DL950™, Dow Chemical, Horgen, Switzerland). A commercial starch derivative was also present at 3 wt%. Coatings were applied to a commercial 80 gm^{-2} woodfree base paper at a speed of 1000 m min^{-1} using a pilot scale coater fitted with a roll applicator blade metering device (Valmet Autoblade™). Coat weights of 14 gm^{-2} were applied to both sides of the paper. The coated papers were then passed through an 11-nip supercalender (Kleinwefers AG, Germany) at a speed of 800 m min^{-1}, a temperature of 100°C, and a linear pressure of 300 kN m^{-1}.

Results from laboratory calendered samples are described in the section on gloss and coating structure and further details of these samples are given in that section. Reflectometric data presented in this paper were obtained using a prototype of the imaging reflectometer during extended trials at Imerys' UK laboratories.

COATING COMPOSITION

A simple use of effective refractive index helps identify the type of pigment used in a coating. However, the method can be ambiguous because even with a particular pigment type, differences in formulation, base sheet, application method, or finishing conditions can lead to significant differences in coating structure.

Figure 1 illustrates the kind of variation in refractive index measured for coatings studies in this work. Notice that calcium carbonate pigments tend to give lower coating refractive index than do clays, even though the refractive index of calcium carbonate (n ~ 1.66) is higher than that of clay (n ~ 1.55). The reason is that calcium carbonates tend to pack with more open structures, thus the coating layer contains relatively more air.

In Fig. 1, some data are shown for representative coatings after offset printing. All the printed papers show a relatively high effective refractive index and differences between the coating types are much less marked. The

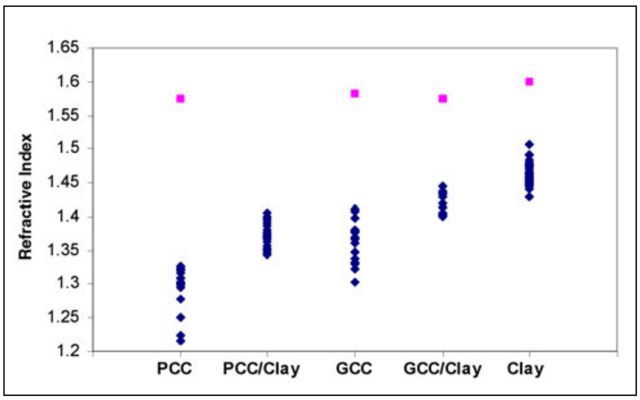

Figure 1 Effective refractive index of paper coatings with various pigment compositions (diamonds). The range of refractive index within each pigment group is due to differences in formulation and application conditions. The pigment type in a coating can sometimes be recognized from the refractive index alone. The squares show the effective refractive index of representative papers from each formulation after printing.

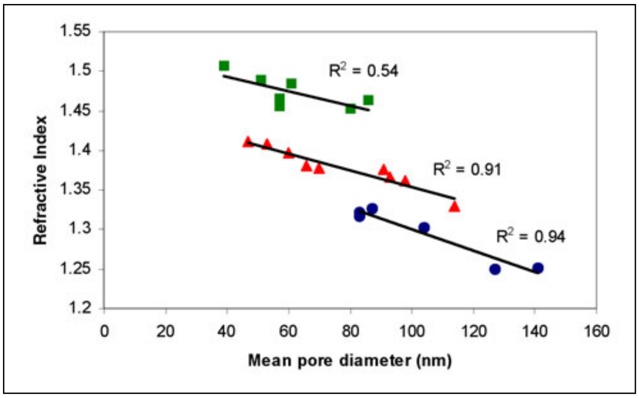

Figure 2a Refractive index versus mean pore diameter (determined by mercury porosimetry) for a range of paper coatings containing various pigment types.

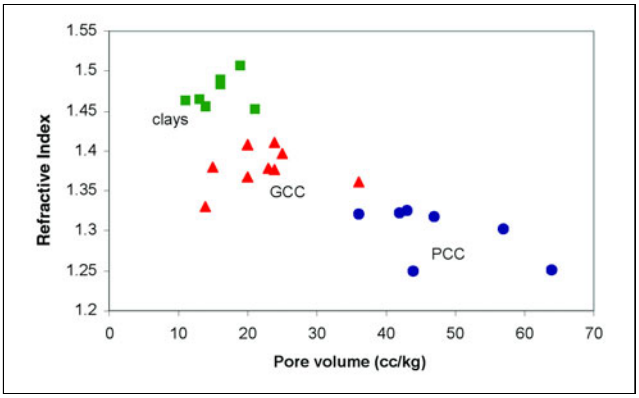

Figure 2b Refractive index versus pore volume for the same coatings.

reflectometry technique is very surface-sensitive (see for example Hiorns, et al. [19]) and in this case the measurements sample only the surface layers of the ink.

COATING STRUCTURE AND POROSITY

Because refractive index is determined by composition and structure, we would expect that the effective refractive index measured by reflectometry should relate to surface porosity. For a range of samples of similar coating chemistry (i.e., similar formulation and pigment type), we would expect differences in effective refractive index between samples to relate directly to differences in particle packing and hence void fraction at the coating surface.

Mercury porosimetry is the usual technique for measuring the pore size and volume of paper coatings. The method is rather laborious and the data analysis is complicated if the volume of the coating layer is to be separated from that of the base sheet [8, 17]. Other authors, such as Gane and Ridgway [20], have carried out mercury porosimetry on coating color tablets and have then used commercially available software (Pore-Comp, University of Plymouth, UK) to correct for compression of the sample. The mercury porosimetry method was applied to the range of coated papers available for this study. Figures 2a and 2b show the relations between effective refractive index and porosimetry pore size and pore volume.

The relationships are quite striking. Within a given pigment type, refractive index correlates extremely well with mean pore diameter (Fig. 2a). Data for pore volume (Fig. 2b) are more scattered, but across the range of pigments used the refractive index generally decreases as pore volume increases (as might be expected). However, within a given pigment group, there is often little correlation between refractive index and pore volume. Indeed, in the case of clays, an apparent "anticorrelation" may be apparent.

One reason for the scatter on the pore volume measurements is simply experimental error in determining the pore volume by mercury porosimetry, which can be relatively large. However, mercury porosimetry and optical reflectometry also sample different regions of the coating layer. The detail of the correlation between them gives insights into the coating structure. Reflectometry is very surface-sensitive for opaque or scattering surfaces. It is difficult to quantitate the interaction volume, but it may be of the order of the microroughness, and recent studies on multilayer coating bear this out [19]. Mercury porosimetry on the other hand is (largely) a bulk measurement technique. The measured pore volume is characteristic of the whole coating layer, including interface regions.

In many cases, the surface of the coating layer may be expected to have a different structure to that of the bulk. Differences may be due to coating color-base sheet interactions, drying, and subsequent calendering, as discussed by Hiorns *et al.* [12]. The "ink-well" model is an example of pore structure that would have no obvious correlation between pore diameter and volume determined by mercury porosimetry (this kind of structure is discussed by Kettle *et al.* in references [21] and [22]).

In Fig. 2a, the three pigment groups obviously form distinct classes. While the correlation between effective refractive index and pore diameter within each group is strong and linear, it is possible that for a given pore diameter (as determined by mercury porosimetry), clays, GCCs, and PCCs each have distinctly different effective refractive indexes. However, we expect that refractive index should relate to surface void fraction (not strictly pore diameter), and therefore pore density is also of importance. The void fraction will be related to the product of pore diameter with pore density.

The importance of surface pore density (number of pores per unit area) has been highlighted in earlier work on ink setting [9]. For a single pore, ink imbibition rate increases with decreasing pore diameter. However, in a coating with many pores, the surface pore density is also crucial in determining the overall imbibition rate. For a give pore diameter, a surface with more pores per unit will absorb a greater volume of fluid per unit time. Clays tend to have relatively low surface pore densities owing to the size, shape, and packing of platy particles at the surface. In contrast, PCCs can have relatively high pore densities owing to their open packing properties. Thus, in Fig. 2a,

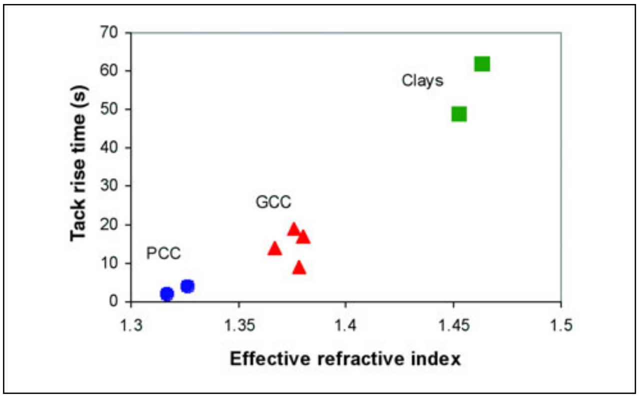

Figure 3 Tack rise versus effective refractive index for pigment coatings having a pore diameter around 80 nm as measured by mercury porosimetry.

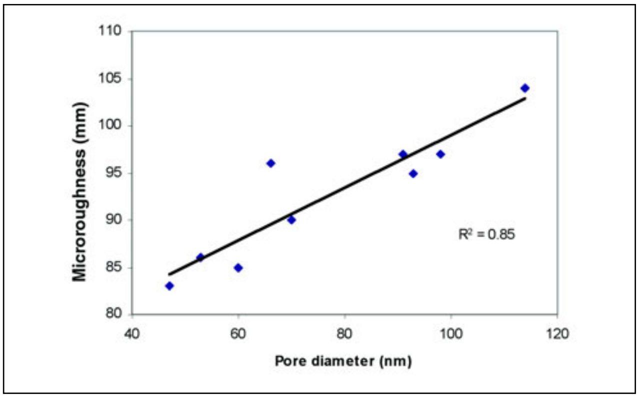

Figure 4 Within a pigment class, microroughness also correlates quite well with pore diameter. Larger pores generally mean greater surface microroughness and lower refractive index due to a greater proportion of air to pigment at the surface. These data are for GCCs.

higher surface pore densities in the PCC coatings can explain why they have the lowest effective refractive index (for a given pore diameter).

We can examine the relation between refractive index (surface porosity) and tack rise time, as measured using the SeGan ink surface interaction tester (ISIT), for coatings of a given pore diameter. For coatings with approximately equal surface pore diameters, we expect the tack rise time to be strongly correlated with the surface pore density. For a given pore diameter, the higher the pore density, the faster the ink vehicle imbibition and hence, the faster the tack rise. The effective refractive index is a measure of surface porosity, but for a given pore diameter (and basically similar surface chemical composition), variations in effective refractive index should relate to variations in surface pore density. Figure 3 shows the correlation between tack rise and effective refractive index for coatings having similar pore diameters (around 80 nm) as measured by mercury porosimetry. Across the range of pigments, the correlation is fair and lends further support to the ideas presented above.

Microroughness also correlates with pore diameter (Fig. 4). Both refractive index and microroughness are indicative of the pigment packing at the surface. In general, more open packing means larger surface pores, which in turn leads to greater surface microroughness and a reduced refractive index.

GLOSS AND COATING STRUCTURE

Gloss is just the reflectance of the surface determined according to some defined standard method. Various standards exist, such as TAPPI 480 om-92 and DIN 54502, which may differ in terms of geometry of illumination, wavelength, angular acceptance, and refractive index of the reference material.

Macroroughness and microroughness can play a significant role in the angular distribution of reflected light. A complete picture of the gloss mechanism is not possible without measurements of both. A purely macrorough surface of some given refractive index simply defines the angular distribution of reflected light. If the angular acceptance of the gloss meter is less than the angular width of the reflected beam, then changes in macroroughness lead to changes in gloss. However, if the angular acceptance of the gloss meter is larger than the angular width of the reflected beam, then macroroughness can change with no apparent change in measured gloss. And further, two gloss meters of different acceptance angle will record different gloss changes.

The effect of microroughness on reflectance is more complicated, depending significantly upon wavelength and angle of incidence, but it always has an important influence on gloss. A knowledge of micro- and macroroughness can be used together with refractive index to determine whether pigment or base sheet effects are responsible for observed gloss variation.

The inter-related effects of microroughness, macroroughness, and refractive index on gloss may be illustrated by data obtained from a laboratory calendering study. Calendering modifies coating structure (see Hiorns *et al.* [12]), producing smoother sheets and creating gloss. A sample of lightweight base paper was pilot coated with a UK kaolin formulation (11 pph SB latex, 3 pph starch, and 0.5 pph calcium stearate), then calendered using a laboratory super calender (Perkins Supercalender) at constant pressure and temperature (1000 psi pressure, 68°C temperature) through varying numbers of nips.

Figures 5 and 6 summarize key results. The first few passes through the calender produced a large increase in sheet gloss. Subsequent passes (up to 15) yield smaller improvements in gloss as measured by the TAPPI standard (Fig. 5). These data are useful in themselves, but do not offer any real insight into what is happening to the coated paper. Reflectometric data (presented in Figs. 6a and 6b) give a much more complete understanding of why the gloss changes as it does. The first few passes through the calender lead to a large decrease in macroroughness (base sheet effects), while microroughness and refractive index (pigment effects) only change slightly. Evidently, the first passes through the calender compress

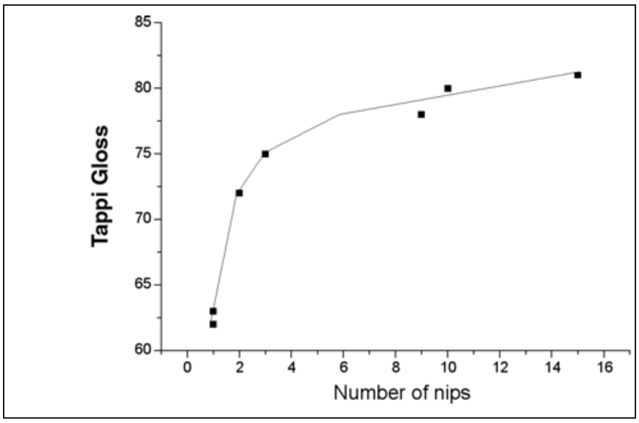

Figure 5 TAPPI gloss as a function of passes through the calender.

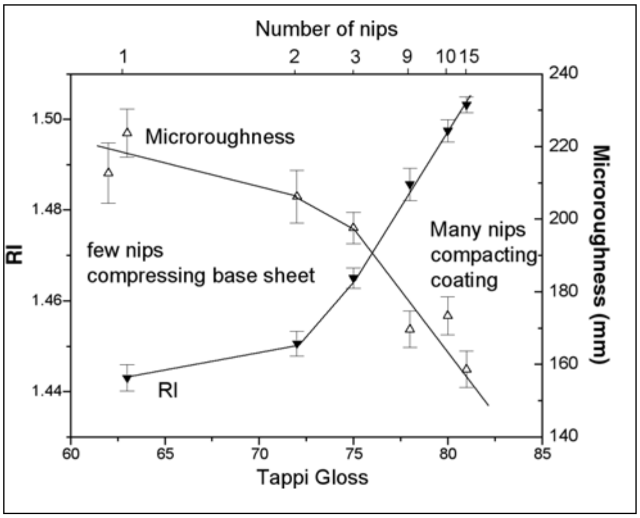

Figure 6a Data from a laboratory calendering exercise showing changes in refractive index and microroughness as a function of number of nips. Paper was pilot coated both sides with a clay formulation. The first passes through the calender compress the base sheet (seen as a large decrease in macroroughness), but produce relatively small changes in refractive index and microroughness. A larger number of nips begins to compress the coating layer, resulting in compaction (increasing RI) and orientation of the clay (decreasing microroughness).

the base sheet fibers, leading to a smoother base sheet. Light is, therefore, reflected from the sheet into a narrower angular range (although the total light reflected may not actually change much). After about nine passes through the calender, the base sheet has reached a point where further smoothing does not occur. Indeed, there is some evidence of a slight roughening at the highest number of nips. However, as the base sheet becomes relatively incompressible, changes in coating structure occur, as manifested by the increase in refractive index and

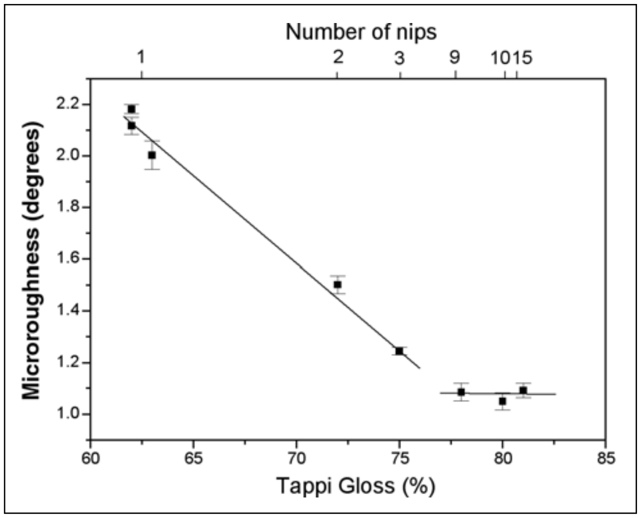

Figure 6b Macroroughness versus number of nips and TAPPI gloss. The first few passes through the calender compress the base sheet and have a significant effect on the macroroughness. The larger number of nips does not affect base sheet smoothness, but gloss continues to rise owing to compaction of the pigment coating.

decrease in microroughness. The coating exhibits a reduction of surface porosity due to compaction and reorientation of clay plates. These physical changes result in decreased microroughness. Both changes in microroughness and refractive index contribute to the continuing rise in gloss observed at large numbers of nips.

As described in Paper I [18], the determinations of effective refractive index, micro- and macro-roughness are largely independent of each other. The model of Eq. 1 can be used to combine these data and calculate the surface reflectance. By integrating Eq. 1 over a suitable range of acceptance angles, we can predict gloss from the roughness and refractive index alone. This calculation has been done for data collected in the calendaring study described above. Figure 7 shows the result. The data are not scaled for the gloss standard used or to take account of instrument geometry. Note the linearity of the relationship between measured and predicted reflectance. This linearity confirms that the changes in refractive index and roughness do indeed provide a largely complete physical description of the gloss mechanism for this calendaring study.

CONCLUSIONS

Analysis of refractive index was found to be an alternative indirect method for inferring pore size and volume within a coating. Within a given pigment type, refractive index correlates extremely well with mean pore diameter. Data for pore volume are more scattered, but across the range of pigments used there is a general trend of refractive index decreasing as pore volume increases. The detail of the correlations is readily explained by considering the different packing structures of the coatings. The role of pore density is also highlighted. For pigment coatings of similar pore diameter, a fair correlation is obtained between effective refractive index and tack rise time.

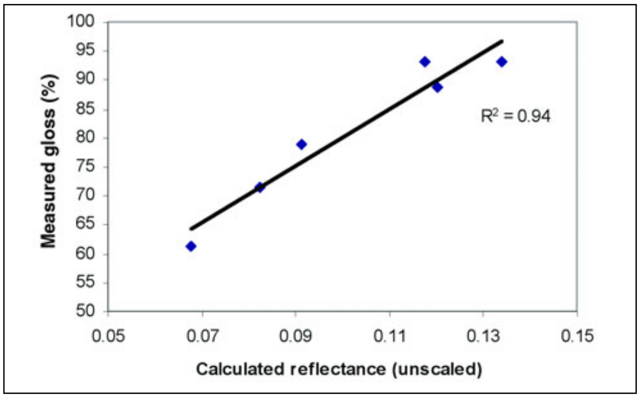

Figure 7 Measured gloss versus reflectance calculated on basis of reflectometric parameters (note the calculated data are not scaled).

A good correlation exists between the microroughness of a set of GCC coatings and the refractive index, indicating the importance of pigment packing at the surface. A more open packing gave larger surface pores, a more microrough surface and a lower refractive index.

The reflectometer has the advantage of simultaneously measuring microroughness and macroroughness. A statistical measure of roughness and an indication of point-to-point variation across a sheet may be obtained in a few tens of seconds. This information can be used, for example, to probe the changes in surface topography that occur during calendering. In this work we found that the first few nips decreased macroroughness, probably due to smoothing of the base paper. A larger number of nips do not affect base sheet smoothness, but gloss continues to rise owing to compaction of the pigment coating, which is evident as a decrease in the refractive index and the microroughness.

The preliminary data presented in this paper show that reflectometry is a useful technique for investigating coating structure and for explaining the origins of gloss. Further work is needed, both to extend the range of applications and materials studied, but also to examine the fundamentals. In particular, the role of diffuse scattering needs further investigation and quantification. Further studies of the relation of refractive index to porosity are also desirable. **TJ**

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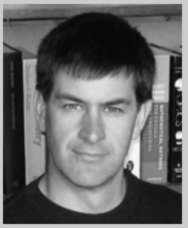
INSIGHTS FROM THE AUTHORS

We had previously used gloss goniophotometry to study coated papers. The method seemed a powerful way of studying paper surfaces, but we were frustrated by the difficulty and slowness of the experimental procedure. We therefore decided to develop instrumentation to make the method much faster, easier, and more accessible. At the same time, we wanted to examine the potential of the new instrument by applying it to a wide range of coated papers. In particular, we were interested in the relation between refractive index and surface porosity (which followed from earlier work on ink imbibition) and on gloss mechanisms.

The hardest part of the research was achieving stable microroughness measurement within the practical constraints of the new instrument design. Whenever new or improved methods of material characterisation are introduced, there are nearly always new things to be discovered. For us, there have been two findings which stand out as particularly interesting. The first is that a simple two-scale roughness model of a paper surface (macroroughness plus microroughness), together with refractive index, is a reasonable predictor of gloss. The second is how well refractive index relates to surface porosity.

Reflectometric measurements may benefit product developers and trouble-shooters by giving direct information on gloss mechanisms in a sheet, while refractive index measurement has the potential to assist understanding of issues like print mottle. Further work is needed on both these aspects. The next steps are to study mapping of refractive index over a sheet surface to correlate with mottle and to examine effects of multiple scatter on refractive index and gloss.



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NEWS ABOUT TAPPI STANDARDS, TIPS, AND TEST METHODS

TAPPI Standards Guidelines revised

The TAPPI Quality and Standards Management Committee has approved a revision to the Regulations and Style Guidelines for TAPPI Standards. The full document may now be downloaded from the web at http://www.tappi.org/content/pdf/standards/tm_guidelines_complete.pdf.

The major change resulting from these guideline revisions is that the language defining TAPPI Standards formerly covered only test methods and the practices related to methods. This language has now been expanded to include specifications, glossaries, and guidelines. Glossaries and guidelines have frequently been a part of TAPPI Technical Information Papers (TIPs), but these new regulations now allow such documents to undergo the rigorous review processes that can make them true TAPPI Standards.

Adding the possibility of writing specifications is a new direction for TAPPI. The regulations clearly state, however, that specifications must be developed in a manner that does not violate the TAPPI Antitrust Policy in any way. We must be certain that specifications are written to be inclusive, strictly voluntary, and to not cause any barriers to trade or restrictions on any company doing business. This will be a new challenge for the TAPPI technical groups. Our aim is to develop specifications that will be helpful to the industries TAPPI serves. Specifications have always been a part the standards of other, such as ASTM and ISO, so issuance of such documents is not unprecedented.

TAPPI Technical Information Papers will continue to be a part of TAPPI's offerings, but some existing TIPs can and should be upgraded to the level of Standards. This will also be a new challenge for our technical experts: to determine what is appropriate as an industry standard and what is not.

To distinguish between the new definition of a "TAPPI Standard" and the definition of TIPs, we have made some terminology changes on the general information page for these documents on the TAPPI web site. Please direct any questions about these changes to Charles Bohanan at cbohanan@tappi.org.

Review Schedule for TAPPI Test Methods

The system for review and development of TAPPI Test Methods requires an announcement of review dates for methods so that any interested TAPPI members may access the drafts and submit comments. The Review Schedule on the web has just been updated to include all of the methods that will be up for review from July 2006 through December 2006.

TAPPI members may comment on methods that are posted for review by going to the home page (www.tappi.org), logging on with your member ID and password, then clicking “My TAPPI.” When your personal page appears, click on the “Test Method Public Draft Review” link on the left-hand side. You do not have to be a member of the Standard Specific Interest Group for any method in order to comment on the draft. If you have joined the Standard Specific Interest Group for any of these methods, you will receive a notice for ballot at the proper time with instructions for casting your vote and making your comments. If you wish to join the Standard Specific Interest Group for a particular method, go to the Subject Category page for links to the sign-up forms.