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FURTHER INVESTIGATIONS INTO SURFACE STRUCTURE
AND THE BIDIRECTIONAL REFLECTANCE DISTRIBUTION FUNCTION

by

Marsha F. Bilmont

A Thesis Submitted to the Faculty of the
COMMITTEE ON OPTICAL SCIENCES (GRADUATE)
In Partial Fulfillment of the Requirements
For the Degree of
MASTER OF SCIENCE

In the Graduate College
THE UNIVERSITY OF ARIZONA

1985
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This thesis has been approved on the date shown below:

[Signature]
William L. Wolfe
Professor of Optical Sciences

Date 11/4/85
To John

Our lives are like the plants
    floating along the water's edge
Illumined by the moon.

Ryōkan
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I would like to take a few paragraphs to mention the people who made this paper possible. Foremost is my advisor, Professor William Wolfe. His support and encouragement through all the stages of this work is deeply appreciated.

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ABSTRACT

The microrough range of surface structure is defined and its effect on light scattering analyzed. The vector theory is used to model scatter for randomly isotropic surface structures. An experiment is presented in which scattering from three reflective surfaces is measured. The scatter for each surface is characterized by the Bidirectional Reflectance Distribution Function and the related surface roughness power spectrum. Results are compared to the vector theory model and non-scatter characterizations of surface structure.
The Bidirectional Reflectance Distribution Function (BRDF) has gained popularity in recent years as a measure of the optical scattering of opaque materials (Hubbs, Brooks, Nofziger, Bartell and Wolfe, 1982; Griner, 1979). Nicodemus first derived the BRDF (1965) as a more general and complete variation on reflectance:

$$\text{BRDF} = f_r = \frac{1}{P_i} \frac{dP_r}{d\Omega_i} \quad [\text{sr}^{-1}]$$  \hspace{1cm} (1.1)

$P_i$ is incident power
$dP_r$ is differential reflected power on the detector
$d\Omega_i$ is differential projected solid angle incident

It has the quality of separating reflectance into its differential elements. Such a function requires that a diffuse scatterer, which reflects equally in all directions, has a constant BRDF at all scattering angles:

$$f_r = \frac{\rho}{\pi}$$

$\rho$ is hemispherical reflectance

Motivation and Objectives

Reflected scatter occurs because ideal surfaces - plane or curved - can not be manufactured. Even a highly polished substrate has
been worked with polishing compounds of finite particle size which gouge out a microscopic terrain. Minute variations in material density, pressure in the tools and mechanical stresses on the substrate boundary contribute to the microstructure. A thin coating may smooth out some of the most grave substrate discontinuities beneath it, but also may contribute to deviations from the ideal smooth curve by scratching or particle clumping.

Many size orders of defects result. Figure errors are large deviations measured over the extent of the surface. They contribute to aberrations in the system and therefore lower image quality. Finish errors or microroughness are second order deviations of microstructure, which must be sampled statistically over a very small region of the surface (Church, 1983). It is this higher order structure which has first order effect on scatter.

Scatter is usually associated with the background noise into an optical system. Infrared systems are often background limited and therefore are particularly sensitive to scatter effects. Lowering or redistributing the scatter in a system can lead to a substantial improvement in the output. In turn, understanding the relationship between microstructure and the distribution of scattered light, as well as polarization and coherence effects and wavelength and incident angle dependence may result in better control of scatter through the system. As ever more stringent system requirements are imposed on infrared and visible systems, such control becomes essential.
The first known study of scatter was performed by Rayleigh in the nineteenth century. This was a study of atmospheric effects on sunlight and resulted in the Rayleigh 'blue sky' scatter dependence on wavelength \( f_r \propto \lambda^{-4} \). The theory was based on the scalar diffraction of unpolarized light by widely dispersed particles. Several authors have carried through the scalar theory for a microrough surface (for example, Davies, 1954; Beckmann and Spizzichino, 1963). Surface and optical factors were included.

The use of polarized light required a new approach to scatter theory. Many theorists (see for example Elson and Ritchie, 1971) treated microroughness as a perturbation of an air-plasma boundary. Maxwell's equations were manipulated to determine the power scattered per unit solid angle. Because this method treated light as a vector quantity, it was useful for analyzing polarized light. Elson later refined the theory into an expression for power scattered per solid angle and showed that it also was a product of three terms, the Rayleigh factor, an optical factor consisting of geometric and dielectric variables and a surface-dependent factor (Elson, 1976).

Experimental work has focussed on modeling the scatter of statistically random microrough surfaces. Harvey (1976) demonstrated that BRDF provided a graphical representation of scatter that, when plotted versus a geometrical term on a log-log scale, had surface statistic dependent linear asymptotes. Later Wang (1983) showed that a one-to-one relationship between the BRDF and the surface roughness power spectrum existed.
The ultimate objective of the body of this thesis is to tie up some of the loose ends in the experimental examination of scatter from statistically describable, isotropic, reflecting surfaces. Specifically, it was the author's intent to examine two as yet unexplored topics: scatter out of the plane of incidence, and variations in scatter over a range of surfaces and wavelengths.

Of the two, the former subject was most successfully covered. In-plane and out-of-plane measurements appeared consistent. However, results were inconclusive on the wavelength dependence and raise new questions about the nature of surface structure and the surface roughness power spectrum.

Out of a need to compare the surface roughness power spectrum obtained from BRDF and from more direct profiling of the surface, a substantial amount of time was spent investigating profiling methods. The three most common techniques are by mechanical stylus, interferometric phase measurement and electron microscope evaluation. One instrument implementing each technique was chosen, based primarily on availability and time considerations. Not all proved useful for the types of surfaces examined here. Descriptions of procedures and results for each are included for future reference.
Microroughness characterizes all nominally smooth surfaces. Any such surface can be delineated as the superposition of three functions; the shape or curvature of the ideal surface, long-range waviness or figure error and short-range, random surface structure. The first two functions can often be controlled to reasonable levels during formation of the surface. The last is the microroughness and we denote its function as \( z(r, \theta) \). It is the primary contributor to surface scatter.

Scattering at a Microrough Surface

In theory, any two dimensional function \( z(x, y) \) can be decomposed into infinite sine and cosine functions:

\[
z(x, y) = \int \int Z(p, q)e^{i(px + qy)} \, dpdq.
\]  

(Gaskill, 1978), where \( p \) and \( q \) are wave numbers for components in \( x \) and \( y \) respectively. In fact, we have limited \( p \) and \( q \) by our definition of \( z(x, y) \) to wavelengths less than the diameter, \( D \), of the sample. Church (1983) provides a somewhat arbitrary lower limit of \( 10/D \) for spatial frequencies in \( z(x, y) \). However, surface scattering is restricted to a very fixed range of spatial wavelengths within the microrough domain, as will be shown.
If \( z(x,y) \) consisted of a single spatial frequency component, the surface would have the form of a sinusoidal grating, Figure 2.1:

\[
z(x,y) = z(x) = z_0 \sin \left( \frac{2\pi x}{p} \right).
\]

(2.2)

The scattered light is simply the diffracted peaks. For the case of reflection:

\[
d \left( \sin \theta_i + \sin \theta_d \right) = m\lambda
\]

\( d \) is grating spacing.
\( \theta_i \) is incident beam angle.
\( \theta_d \) is diffracted beam angle.
\( \lambda \) is wavelength of light.
\( m \) is order number.

The azimuthal and x-z planes correspond. The diffracted beam has angle \( \theta_o = -\theta_i \) with the surface normal. Then:

\[
d(\sin \theta_d - \sin \theta_o) = m\lambda
\]

For first order diffraction (\( m = 1 \)):

\[
p = \frac{2\pi}{d}
\]

\[
= \frac{2\pi}{\lambda} (\sin \theta_d - \sin \theta_o)
\]

(2.3)

\[
= k (\beta - \beta_0)
\]

(2.4)

(Wolfe, 1984). The upper limit for spatial frequencies can then be found for \(-10^\circ\) incidence:

\[
(\beta - \beta_0)_{\text{max}} = 1.17.
\]

\[
P_{\text{max}} = 7.37/\lambda \text{ min}.
\]
Figure 2.1. Light Scattering by a Perfect Sinusoidal Grating.
The lower limit is actually a limitation of the scattering apparatus which excludes angles \( \theta_d \) near normal incidence. For \( \theta_d = 4^\circ \), \( \theta_o = -2^\circ \):

\[
P_{\text{min}} = \frac{0.24}{\lambda_{\text{max}}}. 
\]

The spatial frequency range of interest, for scattering at wavelengths between 0.6328 \( \mu \text{m} \) and 10.6 \( \mu \text{m} \) is then:

\[
0.06 \mu \text{m}^{-1} \leq p \leq 20 \mu \text{m}^{-1}. 
\]

It is reasonable to assume that the scatter as a function of wavenumber is proportional to the integral over the diffracted waveform. Many authors have carried through derivations of this problem. Two schools of thought have emerged. One takes the approach outlined above. The result is a scalar, first order approximation to scattering. The second school utilizes electromagnetic theory. This latter approach results in a vector model of scattering due to the polarization vectors of the incident beam.

**The Vector Theory of Scattering**

A model of surfaces which is readily analyzed via electromagnetic theory has been developed. Smooth surfaces of many kinds are formed by processes which are inherently random, although correlation over some small area is expected since repetition is involved in the process. For instance, the particles of a polishing compound have some spatial extent over which gouging of the substrate is approximately uniform. However, we cannot tell exactly where on the substrate each particle will be moved. The surface is assumed to be isotropic since
the particles are uniform and move about the entire surface hundreds of times to complete the process.

The statistical nature of the microroughness assumed, one can determine the rms roughness, \( \sigma \), defined as the standard deviation of \( z(x,y) \). It is found by:

\[
\sigma^2 = \langle z^2 \rangle - \langle z \rangle^2. \tag{2.5}
\]

For a smooth surface, \( \sigma \) is extremely small compared to the dimensions of interest. The function \( z(x,y) \) then consists of many small random perturbations on a plane boundary.

The perturbation model is the basis of the vector theory. The problem becomes a relatively involved boundary value problem (see, for example, Elson and Ritchie, 1971; Hill, 1981). Many forms of solution have emerged. Scatter as a function of angle (BRDF) has the advantage of generality. It is, from a radiometric standpoint, reflectance per scattered solid angle, and therefore independent of incident beam intensity.

The first order term of the BRDF as derived from vector theory has the form:

\[
f_r = F_\lambda \cdot F_0 \cdot F_\delta \tag{2.6}
\]

(Church, Jenkinson and Zavada, 1977). \( F_\lambda \) is the wavelength factor, in the region \( \sigma \ll \lambda \) it has been predicted to be proportional to \( \lambda^{-4} \) the Rayleigh blue-sky factor. \( F_0 \) is an optical factor, a combination of geometric and dielectric parameters. It is the polarization-dependent
factor. For large permittivity, as observed in metallic boundaries, $F_0$ becomes completely geometrical, taking the form:

$$F_0 = \cos \theta_i \cos \theta_s \cdot P. \quad (2.7)$$

$\theta_s$ is polar scattering angle.

$P$ is polarization factor.

For p-p (source-detector) polarization:

$$P = P_{pp} = \left| \frac{\cos \phi_s - \sin \theta_i \sin \theta_s}{\cos \theta_i \cos \theta_s} \right|^2 \quad (2.8)$$

$\phi_s$ is azimuthal scattering angle.

For p-s polarization:

$$P_{ps} = \sin^2 \phi_s / \cos^2 \theta_i. \quad (2.9)$$

For s-p polarization:

$$P_{sp} = \sin^2 \phi_s / \cos^2 \theta_i. \quad (2.10)$$

For s-s polarization:

$$P_{ss} = \cos^2 \phi_s. \quad (2.11)$$

(Wang, 1983). Unpolarized source and detector form linear combinations of the above:

$$P_{su} = \frac{1}{2} (P_{ss} + P_{sp}). \quad (2.12)$$

$$P_{pu} = \frac{1}{2} (P_{pp} + P_{ps}). \quad (2.13)$$

$$P_{uu} = \frac{1}{2} (P_{ss} + P_{sp} + P_{pp} + P_{ps}). \quad (2.14)$$
Note that eq. 2.8 through 2.14 describe three dimensional or out-of-plane scattering effects. Figure 2.2 illustrates the geometry involved.

The last factor in equation 2.6 is the surface factor \( F_g \) commonly known as the surface roughness power spectrum, \( g(x) \) of the microrough function \( z(x,y) \), where:

\[
K = (p^2 + q^2)^{1/2}. \tag{2.15}
\]

To know BRDF \( (\theta_O, \theta_S, \phi_S; \lambda) \) one must transform the two-dimensional spatial frequency as in eq. 2.3 for the one dimensional case. Now:

\[
m^\lambda = d[(\sin \theta_S \cos \phi_S - \sin \theta_O \cos \Phi_O)^2 \n+ (\sin \theta_S \sin \phi_S - \sin \theta_O \sin \Phi_O)]^{1/2}.
\]

The plane of incidence is the azimuthal plane, \( \phi_O = 0 \). For first order \( (m = 1) \) and dropping the subscript on \( \Phi_O \):

\[
w = \frac{2k}{\lambda} [(\sin \theta_S \cos \Phi - \sin \theta_O)^2 \n+ (\sin \theta_S \sin \Phi)]^{1/2}. \tag{2.16}
\]

\[
p = k[(\sin \theta_S \cos \Phi - \sin \theta_O)]. \tag{2.17}
\]

\[
q = k \sin \theta_S \sin \Phi. \tag{2.18}
\]

When \( \Phi = 0 \):

\[
p = k(\beta - \beta_O)
\]

\[
q = 0
\]

and \( w \) reduces to the one dimensional case.
Figure 2.2. Geometry for Out-of-Plane Scatter.
The Power Spectrum

In vector theory, the form of \( g(\mathbf{w}) \) determines \( f_r (\theta_o, \theta_s, \phi; \lambda) \) exactly. It also relates directly to the microroughness as the fourier transform of the autocorrelation function \( G(\tau, \zeta) \):

\[
G(\tau, \zeta) = \langle z(x,y) \, z(x + \tau, y + \zeta) \rangle
\]

\[
G(0,0) = \sigma^2
\]

For an isotropic function \( z(x,y) \), \( G(\tau, \zeta) \) is radially symmetric:

\[
G(\tau, \zeta) = G(\rho)
\]

and:

\[
g(\mathbf{w}) = \int_{0}^{\pi} \int_{0}^{\pi} G(\rho) \, e^{i \mathbf{w} \cdot \mathbf{p}} \, dp \, d\phi
\]

Both \( G(\rho) \) and \( g(\mathbf{w}) \) if known for all values of \( \rho \) or \( \mathbf{w} \) describe the statistics of surface microroughness completely. Table 2.1 lists forms of surface factor and respective autocorrelation which have been suggested (Church, 1983) or observed using several experimental methods of finish evaluation, including mechanical and optical profilometry (Elson and Bennett, 1979; Bennett and Dancy, 1981, Wyant, Koliopolis, Bhushan and George, 1984), electron micrograph analysis (Harvey, 1976; Rasigni, Rasigni, Palmeri and Llebaria, 1981; van Heereveld, Sikkens and Boose, 1985) and light scattering analysis (Elson, Rahn and Bennett, 1983; Wang, 1983).

Equation 2.22a in Table 2.1 lists the one-dimensional equivalent of the exponential power spectrum, found by integrating the autocorrelation in one direction only. This is common of one-dimensional profiling.
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<th>Autocorrelation Function</th>
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<td>$g(\mathbf{n})$</td>
<td>$G(\rho)$</td>
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2-dimensional Lorentzian

$$\frac{2\pi \sigma^2 \delta^2}{[1 + \kappa^2 \delta^2]^{3/2}}$$

$$\sigma^2 e^{-|\rho|/\delta}$$

(2.20a) (2.20b)

2-dimensional Gaussian

$$-\frac{\kappa^2 \delta^2}{2}$$

$$\pi \sigma^2 \delta^2 e$$

$$\sigma^2 e$$

(2.21a) (2.21b)

1-dimensional Lorentzian

$$\frac{2\sigma^2 \delta}{1 + \kappa^2 \delta^2}$$

$$\sigma^2 e^{-|\rho|/\delta}$$

(2.22a) (2.22b)
techniques. BRDF measurements in-plane (Wang, 1983) exhibit this type of power spectrum if the wavelength factor $F_\lambda$ is assumed to be proportional to $\lambda^{-3}$ accounting for the planar nature of the scattering measurement. If this is the case, one might expect that out of plane BRDF takes the form of the two-dimensional lorentzian surface factor, equation 2.20a.

The quantity $\delta$ in Table 2.1 is the correlation length. It acts as a cutoff value for the autocorrelation, since:

$$G(\delta) = \frac{1}{e} \cdot G(0)$$

The values of $\sigma$ and $\delta$ then determine completely the nature of scattering, if the probability law or laws governing $z(x,y)$ are known.
CHAPTER 3

THE EXPERIMENT

In order to complete the objectives of Chapter 2, including observations of scatter over a wide range of surface roughness, some novel sources of samples were needed. Substrates were of two types: polished flats available off-the-shelf, and commercially-made glasses. The commercial glasses were float glass and non-glare glass.

Float glass is produced by pouring molten sand over molten metal and tempering the two. Nonglare glass is float glass that has been dipped in a hydrochloric acid bath, thereby generating a visibly rippled surface. Both float and nonglare glasses are rougher than the typical polished substrates used in optical applications. In particular, nonglare glass showed promise of testing the theory at its limit, \( \sigma \approx \lambda \).

Samples were checked against large pits or scratches, both centers of high, localized scattering effects. The commercial glass substrates were tested over a large area using a DEKTAK Surface Profile Measuring System. An estimate of \( \sigma \) was determined from approximately 100 sampled values across a 1 mm line at 1/100th mm intervals. The DEKTAK proved destructive on metallic coatings but relatively innocuous to glass. Nonetheless the polished substrates were not measured on this instrument to avoid even microscopic damage.

Figure was not important to the scattering measurements because the lower spatial frequency limit of the instrument, calculated in
Chapter 2, was much less than the width of the sample. Low figure seemed desirable, however, to eliminate the need to filter out low spatial frequencies from profile data. The figure was measured over the diameter of the sample, in our case the beam diameter, 1 cm. For all samples, the figure varied as a function of position on the sample and had values ranging from \( \lambda/2 \) to \( 16\lambda \) in the visible for commercial glasses as measured by the DEKTAK. The figure of the polished substrates was observed interferometrically and had values on the order of \( 1\lambda \).

Three samples were selected for further testing. One nonglare piece (NG2), a standard off-the-shelf mirror (SM1) and a superpolished, fused silica substrate (YW1). SM1 was obtained precoated. It was chosen on the basis of previous in-plane scattering data which showed it to be a typical example of one-dimensional exponential power spectrum. YW1 was used by Wang in 1983 for in-plane scattering at both wavelengths of interest. A minor scratch in the original coating required that YW1 be recoated for this study. Regardless, it provided a reference point for our in-plane scattering results. YW1 and NG2 were all coated with aluminum using thermal vapor deposition to a depth of less than 100 nm. The coating on SM1 was unknown.

### The Scattering Apparatus

Scatter measurements were made on the Arizona Scattering Machine (AZSCAT) at the Optical Sciences Center's Infrared Laboratory. The apparatus has been described many times (Brooks and Wolfe, 1980; Wang, 1983) and is only treated briefly here to explain departures from other works.
For in-plane measurements, lasers, optics and detector share a common horizontal plane, the plane of incidence. The sample lay in an orthogonal plane and was bisected by the plane of incidence. The yaw angle, $Y$, determined the orientation of the sample plane about a vertical axis through the sample. The angle of the specular beam, $\theta_O$, and $Y$ were equivalent. The detector swung on an arm independent of the sample mount. The polar scattering angle intersected by the detector was:

$$\theta_S = D - Y,$$

where $D$ was measured from the incident beam.

Out-of-plane measurements were achieved by pitching the sample plane back at an angle $P$ from vertical (Figure 3.1). The sample was rotated about its intersection with the plane of incidence to ensure the beam would always be incident at the same spot. The new polar angles are:

$$\theta_O = \cos^{-1}(\cos Y \cos P). \quad (3.1)$$
$$\theta_S = \cos^{-1}(\cos (D-Y) \cos P). \quad (3.2)$$

The azimuthal scattering angle completes the transformation equations.

$$\phi = 180 - \sin^{-1} \left[ \frac{\sin Y}{\sin \theta_O} \right]$$
$$- \sin^{-1} \left[ \frac{\sin (D-Y)}{\sin \theta_S} \right]. \quad (3.3)$$
Figure 3.1. AZSCAT Sample Mount and Detector Configuration. S is Sample.
for zero pitch $\phi$ reduced to $0^\circ$ as expected for in-plane measurements.

The relationships between $\theta_o$, $\theta_s$ and $\phi$ and $Y$, $D$ and $P$ to the sample are shown in Figure 3.2.

The samples were measured at $10^\circ$ yaw and $0$, $2$, $5$ and $10^\circ$ pitch, except NG2, which, because of its high scatter, was measured at $0$, $5$, $10$ and $20^\circ$ pitch. For each run, the detector was moved in logarithmic increments over a $160^\circ$ path, with the smallest increments nearest the specular beam.

The detector path was not symmetric about specular. For in-plane measurements, one portion $-80^\circ < \theta_s < 10^\circ$ was completely back scatter, while the other, $10^\circ < \theta_s < 80^\circ$ was forward scatter. Each of the two segments of the detector path completely sampled scatter from specular out to the sample plane. The two segments were denoted as paths B and F.

Differentiation between forward and back scatter became more difficult for out-of-plane geometries. Forward scatter was scatter into the region bounded by the sample and the plane containing the specular beam and the Y-axis as illustrated in Fig. 3.2. The transition from back scatter to forward scatter occurred when the detector path intercepted that plane. As pitch increased, this happened further out from specular on the F path.

In order to cover a wide range of spatial frequencies, two lasers operating at visible and infrared wavelengths were required. We used a standard HeNe at $0.6328$ $\mu$m and an air-cooled, 7-Watt California Laser Model 8-5500-T-P at $10.6$ $\mu$m. Both lasers were polarized parallel
Figure 3.2. Geometry for Scatter Measurements.
Detector Plane Crossing Plane of Incidence.
Inset: Detector Plane Demonstrating Backscatter and Forward Scatter Regions.
to the plane of incidence. The detectors were a silicon photodiode for
the visible and liquid nitrogen-cooled mercury-cadmium-telluride photo-
conductor for infrared. Neither was polarized. The result was an su
polarization configuration.

The AZSCAT machine yielded values of \( f_r (Y,D,P; \lambda) \). These were
transformed using equations 3.1 through 3.3 into \( f_r (\theta_0, \theta_S, \phi; \lambda) \).
Previous in-plane BRDF measurements had been plotted versus \((\beta - \beta_0)\) (or
\(\sin \theta_S - \sin \theta_0\), "the distance of the observation point from the
specular beam in direction cosine space" (Harvey, 1976). The direct
proportionality of this quantity to one-dimensional spatial frequency
resulted in a simple conversion of BRDF data to a power spectrum format.

In the out-of-plane case (Figure 3.3), the difference in
direction cosines was given by:

\[
|\eta - \eta_0| = \left[ (\alpha \beta - \beta_0)^2 + \gamma^2 \beta^2 \right]^{1\over 2},
\]

\(\alpha = \cos \phi; \ \ \gamma = \sin \phi.\)

The relation to two-dimensional spatial frequency (eq. 2.15) is intact:

\[
\kappa = k |\eta - \eta_0|.
\]

All plots of BRDF in this paper have \( |\eta - \eta_0| \) as the abscissa.

Calibration of incident power, \( P_1 \), on the sample surface
utilized highly diffuse (or lambertian) reference materials with
constant BRDF (eq. 1.2). Combining eq. 1.1 and 1.2:

\[
P_1 = \frac{dP_r}{d\Omega_1} \frac{\pi}{\sigma}\]
Figure 3.3. Geometry in Direction Cosine Space.
For the sample:

\[ f_r = \frac{\partial \frac{dP_s}{d\Omega_i}}{\partial \frac{dP_r}{d\Omega_i}} \]

\( dP_s \) is differential scattered power at the detector.

The voltage output of the detector is proportional to the radiance on the detector:

\[ dP_s = V_s R \cos \theta_s. \]
\[ dP_r = V_r R \cos \theta_r. \]

\( V_s \) is voltage output of the detector with sample in its mount.

\( V_r \) is voltage output with reference in mount.

\( R \) is detector responsivity.

\( \theta_r \) is polar angle of the reference mount.

Then:

\[ f_r = \frac{D}{\pi} \left( \frac{V_s \cos \theta_r}{V_r \cos \theta_s} \right). \]

The hemispherical reflectance of the reference must be known.

A no-reference calibration method was also available. In that case the incident beam was passed through the empty sample mount. The detector arm was rotated such that the beam focused directly on the detector. There were several drawbacks to that method, however.

First, all the incident beam must illuminate the detector uniformly. Second, the beam must be highly attenuated -- to a factor of \( 10^5 \) for the 10.6 \( \mu \)m laser -- to avoid saturation. The beam was passed through a series of tinted and coated glass flats to achieve attenuation, and the attenuating factor of each glass was required. Only one
attenuator was required at near angles for the lambertian reference method. Finally, the emergence of Halon as an excellent diffuse reflector in the visible and of certain gold-coated sandpapers in the infrared reinforced our choice.

Figure 3.4 shows the BRDF of Halon at 0.6328 μm and of 600 grit gold-coated sandpaper at 10.6 μm. Both have very flat profiles out to large scattering angles.

Two artifacts appeared in the BRDF data. These were the instrument profile and instrument noise. The instrument profile resulted from scattering within the AZSCAT machine. The effect was high scatter at small incidence angles, where the detector was not shielded from the internal optics. Shading was added where possible without introducing diffraction effects. For a smooth mirror measured at 10.6 μm, instrument profile exceeded the sample BRDF at scattering angles less than 4° from specular. The instrument noise, or noise equivalent BRDF is given by:

\[ \text{NEBRDF} = \frac{(AB)^{\frac{1}{2}}}{D^*t} P_1 \cos \theta_s \]  

A is detector area.  
B is bandwidth.  
D* is detectivity.  
t is system transmission,  

(Wolfe, 1984). At large scattering angles \( \theta_s \), the \( 1/\cos \theta_s \) term rose rapidly, often overtaking the signal in magnitude.
Figure 3.4. Scatter for Near-Lambertian Reference Materials. 
Paths B (x) and F (o).

a) Halon. HeNe at 10° Incidence.

b) 600 Grit Gold Coated Sandpaper. 
CO₂ at 10° Incidence.
The combined instrument profile and noise at 0.6328 μm are shown in Figure 3.5. The peaks at small and large values of $|\eta - \eta_0|$ effectively limited the scatter measurements to a finite range and ultimately were the limits on spatial frequency. In subsequent data, the instrument profile has been eliminated for clarity.

**Surface Profile Observations**

The final phase of the experiment was to characterize the micro-roughness using a non-scattering mechanism. It was desirable to obtain statistics in the same range of spatial frequencies as the scattering results. Three methods were tried: reducing the original DEKTAK data, optical profilometry and electron microscopy.

The DEKTAK operated by moving the sample horizontally below a low-weight diamond stylus. Vertical motion of the stylus was transformed into electrical impulses to a strip chart recorder. The lateral resolution was limited by the diameter of the stylus tip and the slope of the sample to about 2.5 μm for steep gradients. Because it was a dynamic process, it required careful preparation in leveling the sample to get good vertical resolution. It was very repeatable for measurements at the micron level, but was limited to >1 nm height variation over large gradients. Data were taken manually from the strip charts and reduced using the autocorrelation program listed in Appendix A. As noted previously, only NG2 was tested in this manner.

The optical profilometer used was a WYKO Digital Optical Profiler NCP-1000M. This was a 20X interferometer microscope which averaged surface height over a 0.65 μm spot. The lateral resolution
Figure 3.5. Instrument Profile and Noise Functions. Paths B (x) and F (o).
was thus limited to 0.65 μm. The detector was a 1024 element CCD array. The resolvable rms surface height was 0.3 nm. The data were processed by the instrument computer into surface height statistics, autocorrelation function and power spectrum data.

The NCP-1000M is a Mirau interferometer. It compares the phase of light reflected from a small region of the surface to that from a reference spot. This operation is very similar in principle to that of scattering as discussed in Chapter 2. There are significant differences, however. The source is white light, therefore scatter is incoherent over the working distance of the microscope. In addition, the spatial wavelengths involved in reflection are on the order of the spot diameter. One can assume that measurements are independent of larger spatial wavelengths.

The sample is a 0.67 mm line. Output autocorrelation and power spectrum are one-dimensional. The power spectrum is calculated for the range:

\[ 0.01 \text{ μm}^{-1} < p < 0.769 \text{ μm}^{-1}. \]

where the upper limit is much smaller than the highest spatial frequency of the sample.

Scanning Electron Microscopy (SEM) of the coated surfaces was tried but abandoned since very little or no structure was meaningfully observable on any of the samples. A clean mirror such as YW1 offered very little to focus on. At high magnification, defocus was critical. Even stray dust particles did not provide an object near enough to the
surface to locate it. Further, such particles quickly charged and rendered the surrounding area useless to observation. Some structure was observed on NG2, but the slow varying gradient of the surface was barely visible on the low resolution SEM.

Unlike SEM, Transmission Electron Microscopy (TEM) measurements were performed on a transparent replica of the surface. The lowered resolution of the replica was an acceptable exchange for higher instrument resolution. In addition, the ability to shadow structural features was acquired, enhancing contrast and three-dimensionality of the image.

Sample replicas were obtained from a polymer agent often used to clean mirrors. A thin coat of the cleaning agent was applied to each surface and allowed to dry completely. This coat was stripped off using a piece of Scotch Magic tape for leverage. Once clean, a second coat was applied in the same manner. When dry, the polymer had the consistency of thin cellophane. Tape applied with slight pressure to the second dried coat acted as a sturdy backing to the delicate polymer, without damaging the replicated surface.

An approximately 4 mm² piece was cut from each replica and dusted with 0.22 μm diameter polystyrene spheres. The latter served as references. Carbon-Platinum (C-Pt) shadowing was evaporated onto each replica at a glancing incidence of 85° to a maximum thickness of about 10 nm. A second evaporation of pure carbon backed the C-Pt forming a second, positive replica of the surface.

The C, C-Pt replica was washed in a chloroform bath to remove all traces of the polymer film and tape. Scotch Magic tape did not
dissolve well in chloroform. For future experiments, a substitute should be sought. After two fresh baths, the back of the replica was adhered to a 2 mm diameter, 300 mesh copper grid, ready for measurements.

A Hitachi H500 transmission electron microscope operating at 100KeV was used. Grids were affixed to small, cylindrical mounts and side loaded into the imaging cylinder of the machine. An image could be projected onto a small stage at the bottom of the cylinder or directly onto film below the stage.

The loading mechanism did not allow for tilting the sample. This would have been desireable for stereoscopic viewing. Instead of tilt, translation was used, with one image translated one-third of a photo-frame from the other. Pictures were taken at 42,000X and 70,000X. Results are discussed in Chapter 4.
CHAPTER 4

DATA AND RESULTS

This chapter includes the results of reflective scatter and direct structural examination of three surfaces. The data were presented in forms which facilitated understanding of scattering as well as collating scatter and nonscatter interpretations of the surface structure. The latter was effected by calculation of values of the surface roughness parameter $\sigma$ and correlation length $\delta$ from scatter and structure data.

Scrutiny of the scatter data directly enabled observation of scattering mechanisms. The relationships between in-plane and out-of-plane scatter and between forward and back scatter were best documented in this way. Structural information was also obtained. A ratio of BRDF values at different wavelengths provided a method of calculating $\delta$.

Determination of a surface power spectrum model from the scatter data afforded a direct mechanism for comparison of scatter to surface height profiles. The profile data were, within instrument resolution, one-dimensional, while the scatter was measured from a finite area of the sample. Direct comparison of the power spectrums evolving from the one- and two-dimensional methods would be erroneous. However, since the surfaces appeared to be isotropic, the one- and two-dimensional autocorrelation functions were assumed equivalent. Evaluation of the power spectrum model was made on this basis.
Reflective Scattering Measurements

BRDF Data

Figure 4.1 presents graphical representations of the scatter from sample YW1 along the detector paths B and F as shown in Figure 3.2. The data were plotted as BRDF ($\Theta_0$, $\Theta_S$, $\Phi$; $\lambda$) versus $|\eta - \eta_0|$. For this sample the scatter was symmetric over the two paths with respect to $|\eta - \eta_0|$. Two wavelengths were represented in each plot: 0.6328 $\mu$m (HeNe) and 10.6 $\mu$m (CO$_2$). Out-of-plane data at three pitch angles are indicated. In-plane scatter was 1.5 times higher than out-of-plane over a three order of magnitude range of values at 0.6328 $\mu$m. At 10.6 $\mu$m this factor ranged between 1 and 2. The data became indeterminate at large values as the signal fell below the instrument noise.

Scatter measurements of sample SM1 were made over the same geometric region as YW1. The BRDF is shown in Figure 4.2. The magnitude of scatter was about 15 times that of YW1. The 10.6 $\mu$m curve had zero slope out to an $|\eta - \eta_0|$ value of 0.3.

The out-of-plane curves had a peculiar asymmetry to them. While back scatter had a constant slope, a factor of two lower than the in-plane values, transition to forward scatter on path F exhibited a jump in value of about 4 times, then continued at the same slope as in-plane scatter.

The last sample, NG2 (Figure 4.3), had significantly different BRDF than the first two. Scatter was 5 orders of magnitude higher than for YW1 at small $|\eta - \eta_0|$. Slope varied widely, from near zero to -4 at 10.6 $\mu$m. At large values of $|\eta - \eta_0|$ scattering decreased rapidly.
Figure 4.1. Scatter for Sample YW1.  
0° (O), 2° (I), 5° (V), and 10° (X) Pitch.
Figure 4.2. Scatter for Sample SM1.
$0^\circ$ (O), $2^\circ$ (I), $5^\circ$ (V), and $10^\circ$ (X) Pitch.
Figure 4.3. Scatter for Sample NG2.
0° (O), 2° (I), 5° (V), and 10° (X) Pitch.
The 0.6328 μm curve had a slight upturn in slope due to the instrument noise function.

Relationship Between Scatter and Structure

Wang (1983) instituted a method for calculating the correlation length, $\delta$, from BRDF data at two wavelengths. A major assumption, that the power spectrum had a one-dimensional lorentzian form for all values of $\kappa$ (eq. 2.22a), was made in that work. Then:

$$ f_r (\Theta_i, \Theta_s; \lambda) = \frac{2}{\pi} k^3 F_0 \frac{2\sigma^2\delta}{1 + \kappa^2\delta^2}. \quad (4.1) $$

The technique employed a ratio of $f_r$ at each wavelength and a fixed $|\eta - \eta_0|$ value (with $\Phi = 0$). Then, denoting the correlation length as $\delta_1$:

$$ \delta_1 = (R/R_\lambda^3 - 1)^{1/2} \cdot Q, \quad (4.2) $$

where:

$$ R = f_r (\Theta_i, \Theta_s; \lambda_1)/f_r (\Theta_i, \Theta_s; \lambda_2) $$

$$ R_\lambda = \lambda_2/\lambda_1 $$

$$ Q = \lambda_1\lambda_2/[2\pi \eta - \eta_0 |(\lambda_1^2 - \lambda_2^2)^{1/2}|]. $$

If in fact a two-dimensional lorentzian or gaussian power spectrum (eqs. 2.20a, 2.21a) were present, we would expect:

$$ \delta_2 = [(R/R_\lambda^4)^{2/3} - 1] \cdot Q \quad (4.3) $$

and:

$$ \delta_g = [2\ln(R/R_\lambda)]^{1/2} \cdot Q \quad (4.4) $$
respectively. Details of these calculations are listed in Appendix B. Values of correlation length were determined for each model. Each was calculated for \(|\eta - \eta_0| = 0.1\) and \(|\eta - \eta_0| = 0.3\). The average for each sample and model is listed in Table 4.1.

<table>
<thead>
<tr>
<th>Power Spectrum Models</th>
<th>1-D Lorentzian</th>
<th>2-D Lorentzian</th>
<th>Gaussian</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG2</td>
<td>47.2</td>
<td>36.3</td>
<td>41.2</td>
</tr>
<tr>
<td>YW1</td>
<td>18.0</td>
<td>17.5</td>
<td>4.2</td>
</tr>
<tr>
<td>SM1</td>
<td>5.2</td>
<td>6.6</td>
<td>4.9</td>
</tr>
</tbody>
</table>

Matching a Power Spectrum to the Data

Equation 4.1 gave a direct relationship between BRDF and one-dimensional power spectrums. From it we can plot the data in power spectrum form:

\[ g(\kappa) = \frac{\pi}{r} f_r (\theta_i, \theta_s; \lambda) / (k \kappa F_0) \]

This is shown versus \(\kappa\) for sample YW1 in Figure 4.4. A matched power spectrum curve is included.

Some distinct features were evident in this plot. For small \(\kappa\) the data exhibited very little or no slope. This was characteristic of 10.6 \(\mu\)m data. As \(\kappa\) increased, the curve rolled off to a slope of \(-2\) on
Figure 4.4. 1-Dimensional Lorentzian Power Spectrum for Sample YW1. In-Plane Data. Detector Path F. Model Curves are Ratio (R) and Asymptotic.
a log-log scale for 0.6328 μm data. The data showed great continuity between the two wavelengths, if the upturn of the instrument noise terms were ignored.

In keeping with the power spectrum models of Table 2.1, we sought models exhibiting dependence on σ, δ and κ only. We could have taken the value found using the ratio technique, Table 4.1. Another method of solving for δ, assuming a lorentzian curve, found the asymptotes of the power spectrum plot at large and small κ. From equation 2.22a:

\[ g(\kappa) = 2\sigma^2\delta \quad \kappa \ll 1/\delta \]
\[ = 2\sigma^2/\delta \kappa^2 \quad \kappa \gg 1/\delta \]

At the intersection of the two:

\[ \delta = 1/\kappa. \quad (4.5) \]

A value for σ can be found from:

\[ \sigma = (g(\kappa)/2\delta)^{1/2} \quad (4.6) \]

at either large or small κ.

The out-of-plane data was treated in the same fashion. From equation 2.20a:

\[ g(\kappa) = 2\sigma^2\delta^2 \quad \kappa \ll 1/\delta \]
\[ = 2\sigma^2/\kappa^3 \quad \kappa \gg 1/\delta \]

again:

\[ \delta = 1/\kappa \]
In this case the \( \kappa^{-3} \) dependence indicated a \( -3 \) slope at large \( \kappa \) values on a log-log scale. Finally,

\[
\sigma = \left( \frac{g(\kappa)}{2\pi\delta^2} \right)^{\frac{1}{2}}. \tag{4.7}
\]

The power spectrum, shown in Figure 4.5, was plotted from out-of-plane BRDF data. The fourth power wavelength dependence expected for a two-dimensional power spectrum increased the 10.6 \( \mu \text{m} \) data by a factor of 10 from in-plane data. Two model curves, based on the two methods of calculating \( \delta \) are also shown. In both cases \( \sigma \) was found by equation 4.7.

At 10.6 \( \mu \text{m} \) the curves had near zero slope. As pitch increased, the 10.6 \( \mu \text{m} \) data showed a tendency to roll off at \( \kappa = 0.3 \mu \text{m}^{-1} \). The instrument noise, prominent in the scatter data for this sample (Figure 4.1), would tend to mask this effect, suggesting that the roll-off actually occurred at a slightly lower \( \kappa \) value.

At 2° and 5° pitch, the 0.6328 \( \mu \text{m} \) data had the same \( -2 \) slope as in-plane data. At 10° pitch, a few points around \( \kappa = 4 \mu \text{m}^{-1} \) showed the \( -3 \) slope of the two-dimensional lorentzian curves. The asymptotically-generated curve was determined from these points. The ratio curve did not fit the data well.

The gaussian model was also applied to the out-of-plane data (Figure 4.6). Using \( \delta \) from the ratio technique and from equation 2.21a:

\[
\sigma = \left( \frac{g(\kappa)}{\pi\delta^2} \right)^{\frac{1}{4}} \tag{4.8}
\]

There was good agreement with 10.6 \( \mu \text{m} \) data, but none at 0.6328 \( \mu \text{m} \).
Figure 4.5. 2-Dimensional Lorentzian Power Spectrum for Sample YW1. Out-of-Plane Data. Detector Path F. Ratio (R) and Asymptotic (A) Curves.
Figure 4.6. 2-Dimensional Gaussian Power Spectrum for Sample YW1. Out-of-Plane Data. Detector Path F.
Figures 4.7 and 4.8 are plots of in-plane and out-of-plane power spectrum taken from scattering data for samples SM1 and NG2. The plots are accompanied by the best fit of models described above. Like sample YW1, SM1 (Fig. 4.7) exhibited tendencies of the two-dimensional curves at 10° pitch.

The $\sigma$ calculated for NG2 using 10.6 $\mu$m data was 0.14 $\mu$m. The visible wavelength used was only 5.3 times that. In predicting a theoretical model, we did not use the 0.6328 $\mu$m data to avoid conflicting with the $\sigma \leq \lambda$ constraint. This invalidated the ratio technique for obtaining $\delta$ and eliminated the ability to observe the wavelength dependence of the scatter. The power spectrum of NG2 (Figure 4.8) for in-plane scatter rolled off to a slope of -4. This did not match the roll-off of the one-dimensional lorentzian curve. The out-of-plane data had a single asymptote with -4 slope. The sample appeared to have a lorentzian form, but not of the known models.

Due to the curve-fitting nature of the asymptotically-generated curves, they tended to show better general agreement with the data than the ratio curves. Table 4.2 lists values of $\delta$ found by the asymptotic

<table>
<thead>
<tr>
<th>Power Spectrum Models</th>
<th>1-D Lorentzian $\delta_1$</th>
<th>2-D Lorentzian $\delta_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG2</td>
<td>25.0</td>
<td>11.1</td>
</tr>
<tr>
<td>YW1</td>
<td>11.1</td>
<td>5.3</td>
</tr>
<tr>
<td>SM1</td>
<td>5.0</td>
<td>5.6</td>
</tr>
</tbody>
</table>
Figure 4.7. Power Spectrum Models for Sample SM1. Ratio (R) and Asymptotic (A) Curves.
a) In-Plane


Figure 4.8. Power Spectrum Models for Sample NG2. Asymptotic Curves Only.
method and Table 4.3 lists values of rms roughness found from power spectrum models of Figures 4.7 and 4.8. There was moderate agreement.

A Single Fit Power Spectrum

The BRDF plots in and out of the plane of incidence showed a general tendency to agree in magnitude within a factor of 2. It is possible that the in-plane and out-of-plane power spectrum were also one and the same. Of curves tried, the best fit to all the data for samples YW1 and SM1 was the one-dimensional lorentzian, as shown in Figure 4.9a and 4.9b. This implied a $\lambda^{-3}$ wavelength dependence for out-of-plane scatter, contrary to three-dimensional scatter theories.

The best fit to the 10.6 $\mu$m portion of the NG2 data was the two-dimensional lorentzian (Figure 4.9c and 4.9d). The wavelength dependence could not be determined. Calculating $\sigma$ from equations 4.6 and 4.7 yielded values of 0.14 $\mu$m and 0.09 $\mu$m respectively.

Non-Scatter Surface Structure Measurement

Mechanical Profiles

NG2 was mechanically probed prior to being aluminum-coated. The surface profile and autocorrelation function is depicted in Figure 4.10. NG2, Figure 4.10a, had a relatively smooth varying surface. Very little secondary structure was observed at this scale. The data were sampled at 10-$\mu$m intervals. This appeared sufficient to test a great deal of the primary surface characteristics. The surface roughness, $\sigma$ was calculated from sample values to be 0.15 $\mu$m.
Table 4.3. Values of RMS Roughness from Power Spectrum Models (μm).

Based on Correlation Length Found by Ratio (R) and Asymptotic (A) Methods.

<table>
<thead>
<tr>
<th>Power Spectrum Models</th>
<th>1-D Lorentzian</th>
<th>2-D Lorentzian</th>
<th>Gaussian</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>R</td>
<td>A</td>
<td>R</td>
</tr>
<tr>
<td>NG2</td>
<td>-</td>
<td>1.4x10^-1</td>
<td>-</td>
</tr>
<tr>
<td>YW1</td>
<td>3.7x10^-3</td>
<td>2.6x10^-3</td>
<td>3.9x10^-3</td>
</tr>
<tr>
<td>SM1</td>
<td>8.2x10^-3</td>
<td>8.4x10^-3</td>
<td>6.6x10^-3</td>
</tr>
</tbody>
</table>
Figure 4.9. Best-Fit Power Spectrums.

a) Sample YM1

b) Sample SM1
c) Sample NG2 ($\lambda^{-4}$).

d) Sample NG2 ($\lambda^{-3}$).

Figure 4.9. Continued.
b) Surface Profile

Figure 4.10. Mechanical Stylus Profiles of Sample NG2.
The autocorrelation function for NG2, Figure 4.10b, had very little structure beyond the large peak centered on the origin. One minor spike at 320 μm suggested a stronger correlation between surface heights at that interval. The function was otherwise random, however. The 1/e point occurred in the central peak, yielding a value δ of 25 μm.

Optical Profiles

Figure 4.11 shows surface profiles of the four samples over a 0.65 mm line. All samples were measured interferometrically after scatter measurements were computed, but before replicas of the surface, required for TEM, were made. In effect, these measurements were made of samples in the state closest to scatter measurements. Two of the samples, YW1 and NG2, were also measured using a lower power (10X) objective. The results were comparable, but some differences due to the longer sample length were noted and are indicated in the descriptions. Figure 4.11a shows the optical profile of sample YW1. While σ = 0.54 nm measured over a 0.66 mm sample, it rose to 0.65 nm over 1.3 mm. Sample SM1 appeared more random than YW1, as shown in Figure 4.11b. Average σ was 0.74 nm. It also exhibited the high degree of randomness of YW1. There were some distinct spikes in the data.

Once again sample NG2 proved to have a smooth varying surface (Figure 4.11c). The profile was very similar to that of the mechanical profiler, with very little secondary structure observable. The rms roughness averaged 142 nm over 0.66 mm and 183 nm over 1.3 mm. The
Figure 4.11. Interferometrically Generated Surfaces Profiles.
c) Sample NG2

Figure 4.11. Continued.
dominant structure had a large spatial frequency component on order of the sample length.

The autocorrelation function calculated from optical profile data for each sample is plotted in Figure 4.12. Correlation distances were again calculated as the 1/e point. Values of δ calculated using the 20X objective were considerably lower than those found using the 10X, due to the shorter sampling interval. Table 4.4 compares δ for different sampling intervals.

YW1 and SM1 had predominantly random autocorrelation (Figure 4.12a and b). Both fell off sharply to zero. SM1 was the less random

| Table 4.4. Values of Correlation Length for Four Sampling Intervals (μm). |
|--------------------------|--------------------------|
|                         | Optical Profiler         | Mechanical Profiler |
|                         | 0.65 μm | 1.2 μm | 10 μm | 100 μm |
| NG2                     | 20      | 30     | 20    | 100    |
| YW1                     | 10      | 30     | -     | -      |
| SM1                     | 12      | -      | -     | -      |

of the two. The peaks and valleys had some symmetry about a 330-μm point to point distance.

Sample NG2 had nearly periodic correlation. The smooth curve of Figure 4.12c is in great contrast to Figure 4.10b which appeared highly random. The sampling interval, which was an order of magnitude larger
Figure 4.12. Autocorrelation From Interferometric Profiles.
c) Sample NG2

Figure 4.12. Continued.
for mechanical profiling, had an enormous effect on the outcome. Even at a sampling interval of 1.2 μm, using the 10X objective, some random character was introduced. From zero the autocorrelation had a slow roll-off suggesting a gaussian contribution to the statistical model.

**Electron Microscope Profiles**

In the preceding sections we noted that the primary structure of the samples used in this study had large spatial frequency contributions on order of tenth millimeters. Electron microscopy was capable of lateral dimensions on this order. However, the instrument resolution was insufficient to observe the slow varying gradients of such structure. Furthermore, the lower order structure appeared to wash out during the replication process. For these reasons, TEM was relegated, in this study, to observation of secondary structure.

A stereoscopic pair of images, taken in translation by TEM, is shown in Figure 4.13 for sample NG2. Magnification was 42,000X. The large sphere on the left of each photograph was a 0.22 μm diameter polystyrene sphere used as a surface height reference. The lateral extent of the images was 1.8 μm x 1.5 μm. Three artifacts, large depressions in the surface, were common to all the samples and likely were due to gas bubbles in the polymer replica caused by overheating during carbon vapor deposition. Figure 4.14 shows half of the stereo pair for sample YW1. Sample SM1 was not evaluated using TEM.

A major problem with TEM measurements was the replication process required. For sample NG2 the process proved totally destructive. Several pieces of aluminum coating were pulled off the glass substrate
Figure 4.13. Stereoscopic Pair in Translation: Sample NG2, 42,000X
Figure 4.14. Sample YW1 at 42,000X.
with the first polymer coating. The rough nature of the sample likely
did not permit good adherence of the aluminum to the surface.

In other cases, replication may have had a less apparent but
still dramatic effect on surface structure. The scatter at 0.6328 \( \mu \text{m} \)
was measured after replication was completed on sample YW1. The BRDF
before and after replication is shown in Figure 4.15. The BRDF of YW1
exhibited change, however. The scatter magnitude was increased 5 times
and the roll-off from zero slope occurred at a larger \( |\eta - \eta_0| \) value.

The spatial frequency limitations of the TEM apparatus were
clearly defined. The wire grid, with spacing of 85 \( \mu \text{m} \), obstructed
observation of lower frequency structure. The instrument resolution was
limited by the C-Pt shadowing grain size. At 42,000X the grains, of
diameter on order of 2 nm, were barely visible.

The low vertical definition of the surfaces was such that stereo
measurements were not satisfactory. Evaluation of surfaces on this
order of roughness would best be done using image processing. Such a
procedure would require digitizing the negative by transmission on a
microdensitometer, obtaining a grey scale of slope values using a
reference such as the polystyrene spheres, and converting the slope
information to surface height values. The last process is thoroughly
described by Rasigni et al. (1981). Due to time constraints the
procedure was not undertaken here.

Comparison of Profile and Scatter Results

Values of \( \sigma \) and \( \delta \) determined by the various methods of this
study are given in Table 4.5. Where more than one value was found by a
Figure 4.15. Effect of Replication Techniques on Scatter Performances. Before (o) and After (*) Replication. Sample YW1.
Table 4.5. Comparison of RMS Roughness and Correlation Length Calculated by Five Methods (μm).

*aSample intervals of 10 μm only.

<table>
<thead>
<tr>
<th></th>
<th>DECTAKa</th>
<th>WYKO 20X</th>
<th>AZSCAT 1-D</th>
<th>AZSCAT 2-D</th>
<th>AZSCAT Gaussian</th>
</tr>
</thead>
<tbody>
<tr>
<td>YW1</td>
<td>-</td>
<td>5.4 x 10^{-4}</td>
<td>2.3 x 10^{-3}</td>
<td>9.6 x 10^{-4}</td>
<td>2.3 x 10^{-2}</td>
</tr>
<tr>
<td></td>
<td>-</td>
<td>20</td>
<td>19.0</td>
<td>18.8</td>
<td>2.7</td>
</tr>
<tr>
<td>SM1</td>
<td>-</td>
<td>7.7 x 10^{-4}</td>
<td>8.4 x 10^{-3}</td>
<td>5.6 x 10^{-3}</td>
<td>1.9 x 10^{-2}</td>
</tr>
<tr>
<td></td>
<td>-</td>
<td>12</td>
<td>5.7</td>
<td>6.4</td>
<td>2.3</td>
</tr>
<tr>
<td>NG2</td>
<td>1.73 x 10^{-1}</td>
<td>1.42 x 10^{-1}</td>
<td>1.4 x 10^{-1}</td>
<td>5.0 x 10^{-2}</td>
<td>3.3 x 10^{-2}</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>25</td>
<td>24.1</td>
<td>24.1</td>
<td>9.1</td>
</tr>
</tbody>
</table>
single method, results were averaged. The results offer some support to the scatter models, but inconsistencies remain.

For the samples, YW1, SM1 and NG2, $\delta$ was very consistent between scatter and non-scatter methods. The surface roughness did not compare as well. The roughest sample, NG2, had the best agreement between methods. The two-dimensional interpretation of the power spectrum yielded slightly lower values of $\sigma$ than the one-dimensional. The Gaussian model gave slightly higher values.

The autocorrelation function of NG2 (Fig. 4.12c) displayed some gaussian tendency over small point separation. The scatter power spectrum showed some tendency toward gaussian, especially in the rapid decline of data at both wavelengths. There was some discrepancy in the correlation length calculated using the two methods, however, suggesting a second contribution to the structural model.

Because of its large surface roughness, some unique characteristics were expected from NG2. The scatter curves (Fig. 4.4) exhibited very high scatter centered not on the incident plane, but on the plane through which the detector passed closest to the specular beam. The profile curves may explain why scatter near specular, rather than back-scatter, predominated. The average peak-to-peak height was 0.3 $\mu$m, while typically distance between peaks was on order of 100 $\mu$m. Therefore, the average slope was about 0.003. This was enough to diverge the incident beam, but with very low large-angle scatter. This was prominent for 0.6328 $\mu$m scattering data.
At 10.6 μm, $\sigma < \chi$ and we would expect the theoretical prediction of scatter to take effect. To some extent it did; the scatter was extended over larger angles. However, the high scatter peaks close to specular still appeared in the out-of-plane data, suggesting a change in the geometrical factor of the vector model.

Sample SM1 also exhibited a significant trend toward higher forward scatter out-of-plane. In context with the diffraction grating example of Chapter 2, such directional scattering was reminiscent of a blazed grating, which directs normally incident light into orders rotationally displaced a fixed angle. The profile data did not exhibit a strong tendency toward such structure, however.
CHAPTER 5

CONCLUSIONS

Both scatter and profile measurements were performed on reflective surfaces with a broad range of structural characteristics. The most persuasive results were therefore the most general. Some effects unique to a given sample were only briefly touched on in this work and should be explored further.

The most significant observation of this study was that BRDF measured out of the plane of incidence had similar form and magnitude to that measured in the plane. In some cases, the out-of-plane power spectrum did show some tendency toward a two-dimensional surface model, different from the one-dimensional model for in-plane. That tendency did not appear with 5° of the plane of incidence, however.

Future measurements should concentrate on rougher surfaces, between 10.0 and 50.0 nm rms, and on wavelengths between 0.6328 μm and 10.6 μm. Both recommendations would facilitate observations around \( \kappa = 0.4 \ \mu m^{-1} \).

There was some contrary evidence of preferential scattering out-of-plane. NG2, the sample with large surface roughness, demonstrated higher scatter out-of-plane near the specular beam. SM1 had large forward scatter out-of-plane. Neither of these tendencies was consistent from sample to sample.
Sample NG2 also demonstrated some effects of large surface roughness on the scatter. One was a rapid decline from high scatter near specular. Another was the tendency as the wavelength decreased toward broadening of the scatter near specular. The latter resulted from a decline in the effect of the surface's correlation. The preferential scattering out-of-plane may have been a third effect; however it occurred with equal magnitude at both 0.6328 μm and 10.6 μm, the latter at least 100 times σ and therefore generally accepted to be within the realm of theory.

Comparison of scatter to non-scatter methods of structural analysis was positive, especially in determination of correlation length. The AZSCAT machine did appear to filter out information from the surfaces of spatial wavelength greater than 100 μm. Inconsistencies were apparent in the determination of σ.

The TEM measurements were of little value to our comparisons. In the future, this method could be a viable source of high spatial frequency information if the difficulties, discussed in Chapters 3 and 4, were resolved. As surfaces become smoother and primary surface structure smaller, this information could be a useful supplement to other forms of observation.
APPENDIX A

PROGRAM TO CALCULATE AUTOCORRELATION FUNCTION

100 REM PROGRAM TO CALCULATE SURFACE FACTOR, \( g(k) \) FROM SURFACE
110 REM PROFILE DATA
120 REM
130 INIT
140 PAGE
150 DIM Z(200), G(300), T(301), U(301), P(301)
151 DATA -2.6, -3.2, -1.5, -1.3, -1.6, -2.1, 0.8, 0.1, -0.5, -0.7, -2.1, -1.3, -1.1
152 DATA -0.4, 0.0, -3.3, -3.6, -4.5, -3.4, -2.8, -1.8, -2.2, -1.6, -2.3, -0.9
153 DATA 1.2, -1.7, 2.2, 0.7, 0.3, 0.4, -0.2, -0.9, -0.6, -2.7, -3.0, 7.1, 4.0, 9.0, 0.8
154 DATA 0.0, -0.4, -0.7, -3.0, -1.9, 2.2, 0.5, 1.6, 0.5, -0.7, -3.6, -1.8, -0.8, 0.5
155 DATA 0.3, -0.5, -0.8, -0.5, 1.1, 1.7, 0.2, 1.9, -0.2, -0.7, 0.3, 2.1, 1.8, 1.2
156 DATA 0.5, 2.1, 1.3, 0.4, 0.2, 0.1, 0.1, 0.3, 0.9, 0.8, 0.3, 2.2, 2.8
157 DATA 0.0, -0.4, -0.7, -3.3, -3.6, -4.5, -3.4, -2.8, -1.8, -2.2, -1.6, -2.3, -0.9
158 DATA 1.2, -1.7, 2.2, 0.7, 0.3, 0.4, -0.2, -0.9, -0.6, -2.7, -3.0, 7.1, 4.0, 9.0, 0.8
159 DATA 0.0, -0.4, -0.7, -3.0, -1.9, 2.2, 0.5, 1.6, 0.5, -0.7, -3.6, -1.8, -0.8, 0.5
160 PRINT "SURFACE DATA ON FILE "
170 INPUT Q$
180 IF Q$="N" THEN 210
190 GOSUB 1110
200 GO TO 240
210 GOSUB 2020
220 GOSUB 4076
230 GOSUB 3030
240 GOSUB 5030
250 END
1000 REM
1010 REM
1020 REM F FILE NUMBER
1030 REM N$ FILE NAME
1040 REM Z<J> SURFACE HEIGHT DATA
1050 REM N OF DATA POINTS
1060 REM V VARIANCE OF SURFACE HEIGHT DATA
1070 REM M1 MEAN VALUE OF SURFACE HEIGHT
1080 REM S 2ND MOMENT OF SURFACE HEIGHT
1090 REM U$ UNITS OF Z
1100 REM
1110 PRINT "FILE NUMBER"
1120 INPUT F
1130 FIND F
1140 INPUT G33:H$
1150 INPUT G33:U$
1160 INPUT G33:N
1170 INPUT G33:U
1180 INPUT G33:M1
1190 INPUT G33:S
1200 PRINT "FILE NAME IS", H$
1210 PRINT "IS THIS WHAT YOU WANT"
1220 INPUT Q$
1230 IF Q$="N" THEN 1110
1240 FOR J=1 TO N
1250 INPUT G33:Z<J>
1260 NEXT J
1270 INPUT G33:U$
1280 INPUT G33:T0
1290 INPUT G33:G1
1300 INPUT G33:G$
1310 INPUT G33:F$
1320 FOR I=1 TO N-1
1330 INPUT G33:G<1>
1340 NEXT I
1350 RETURN
2000 REM SUBROUTINE DATA CALC
2010 REM SUBROUTINE DATA RETRIEVE
2020 REM
2030 REM FILE NUMBER
2040 REM N$ FILE NAME
2050 REM Z<J> SURFACE HEIGHT DATA
2060 REM N OF DATA POINTS
2070 REM V VARIANCE OF SURFACE HEIGHT DATA
2080 REM M1 MEAN VALUE OF SURFACE HEIGHT
2090 REM S 2ND MOMENT OF SURFACE HEIGHT
2100 REM U$ UNITS OF Z
2110 REM
2120 PRINT "FILE NUMBER"
2130 INPUT F
2140 FIND F
2150 INPUT G33:H$
2160 INPUT G33:U$
2170 INPUT G33:N
2180 INPUT G33:U
2190 INPUT G33:M1
2200 INPUT G33:S
2210 PRINT "FILE NAME IS", H$
2220 PRINT "IS THIS WHAT YOU WANT"
2230 INPUT Q$
2240 IF Q$="N" THEN 1110
2250 FOR J=1 TO N
2260 INPUT G33:Z<J>
2270 NEXT J
2280 INPUT G33:U$
2290 INPUT G33:T0
2300 INPUT G33:G1
2310 INPUT G33:G$
2320 INPUT G33:F$
2330 FOR I=1 TO N-1
2340 INPUT G33:G<1>
2350 NEXT I
2360 RETURN
2810 REM
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2020 M1=0
2030 S=0
2040 PRINT "# OF DATA POINTS ";
2050 INPUT N
2060 PRINT "SCALE FACTOR ";
2070 INPUT S8
2080 PRINT "DATA UNITS (MICROMETERS=M; ANSTROHS=A)");
2090 INPUT U*
2100 PRINT "INPUT SURFACE HEIGHT VALUES NOW".
2110 FOR J=1 TO N
2120 READ Z(J)
2130 Z(J)=Z(J)*S8
2140 S=S+Z(J)^2
2150 M1=M1+Z(J)
2160 NEXT J
2170 S=S/N
2180 M1=M1/N
2190 M2=M1^2
2200 V=S-M2
2210 PRINT "VARIANCE","MEAN","<Z^2","RMS SURFACE HT."
2220 PRINT V,M1,S^0.5
2230 REM SUBTRACT MEAN FROM DATA. NEW DATA HAS MEAN OF ZERO
2240 REM
2250 FOR J=1 TO N
2260 Z(J)=Z(J)-M1
2270 NEXT J
2280 RETURN

2900 REM SUBROUTINE DATA-STORE
2910 REM
2920 PRINT "DATA TAPE,FILE TO STORE: ";
2930 INPUT T*,F
2940 PRINT "FILE NAME ";
2950 INPUT N*
2960 IF U*="H" THEN 3100
2970 U$="ANGSTROHS"
2980 GO TO 3110
2990 U*="MICRONS"
3000 IF F=0 THEN 3300
3010 FIND F
3020 MARK I,N*40
3030 FIND F
3040 PRINT T33:N*
3050 PRINT T33:U*
3060 PRINT T33:M1
3070 PRINT T33:S
3080 FOR J=1 TO N
3090 PRINT T33:Z(J)
3100 NEXT J
3110 RETURN

3800 REM SUBROUTINE AUTO GETS AUTOCORRELATION FUNCTION G(I) FROM Z(I)
3810 REM T0 MINIMUM DATA INTERVAL
3820 REM V$ UNITS OF T0
3830 REM G(I) AUTOCORRELATION FUNCTION
3840 REM
3850 PRINT "INPUT MIN DATA INTERVAL T0,UNITS";
3860 INPUT T0,V$
3870 GO=0
3880 PRINT "INPUT MIN DATA INTERVAL T0,UNITS";
3890 INPUT T0,V$
3900 FOR I=1 TO N-1
3910 G(I)=0
3920 FOR J=1 TO N-1
3930 G(I)=G(I)+Z(J)*Z(J+I)+G(I)
4140 NEXT J
4150 G(I)=G(I)/N
4160 NEXT I
4170 G=0
4180 FOR I=1 TO N-1
4190 IF G(I)=G1 THEN 4210
4200 G=G(I)
4210 NEXT I
4220 RETURN
5900 REM
5910 REM SUBROUTINE AUTOPLOT PLOTS G(T)
5920 REM
5930 VIEWPORT 5,125,5,95
5940 Y9=U
5950 X9=H
5960 X=-X9/5
5970 Y=-Y9/2
5980 PAGE
5990 WINDOW X+1.5,Y9,Y9+1.5,Y9
6000 MOVE 0,Y9
6010 DRAW X9,Y9
6020 DRAW X9,Y
6030 DRAW 0,Y
6040 DRAW 0,Y9
6050 MOVE 0,0
6060 DRAW X9,0
6070 DRAW X9,0
5100 IMAGE 2E
5110 FOR J=0 TO X9 STEP 10
5115 HOVE J,Y9
5120 DRAW J,Y9*0.98
5130 MOVE J,Y
5140 DRAW J,Y*0.96
5150 MOVE J-X9/25,Y*1.2
5160 PRINT J*T0
5170 NEXT J
5180 FOR J=Y9 TO Y STEP -Y9/5
5190 MOVE 0,J
5200 DRAW 0.99*X9,J
5210 NEXT J
5220 FOR I=1 TO N-1
5230 DRAU I,G(I)
5240 NEXT I
5250 C1=Y9/20
5260 MOVE X9/4,Y+1.4
5270 IMAGE 20A,11A
5280 PRINT USING 5330:"DISTANCE ON SURFACE ":U$
5290 L=LEN(U$)
5300 FOR L=1 TO L1
5310 B#=SEG(U$,L,1)
5320 MOVE X+1.3,Y9-1.5-C1*L
5330 PRINT B$
5340 NEXT I
5350 AS="SOR"
5360 FOR L=1 TO 4
5370 B#=SEG(A$,L,1)
5380 MOVE X+1.3,Y9-1.5-C1*(L+1)
5390 PRINT B$
5400 NEXT L
5410 A$="\n"
5420 FOR L=1 TO 4
5430 B#=SEG(A$,L,1)
5440 MOVE X+1.3,Y9-1.5-C1*(L+1)
5450 PRINT B$
5460 NEXT L
5470 MOVE X9/3,2*Y9/3
5480 PRINT "AUTOCOVARIANCE FUNCTION"
5490 MOVE X9/3,2*Y9/3+4
5500 IMAGE 10A,3D,3D,1X,11A
5510 PRINT USING 5580:"RMS SIGMA=":V$+0.5;U$
5520 MOVE X9/3,2*Y9/4
5530 IMAGE 4A,7A,3A,7A,2D
5540 PRINT #1;"TAPE ":T$;FILE ";F
5550 RETURN
6000 REM
APPENDIX B

CALCULATION OF CORRELATION LENGTH FOR TWO-DIMENSIONAL SURFACE MODELS

For a two-dimensional lorentzian type power spectrum (equation 2.20a):

\[
f_r (\theta_1, \theta_2; \lambda) = 2\kappa_0^4 F_0 \frac{\sigma^2 \delta^2}{[1 + \kappa_0^2 \delta^2]^{3/2}} \quad (A.1)
\]

\[
\frac{f_{r_{\lambda_1}}}{f_{r_{\lambda_2}}} = \frac{\kappa_{\lambda_1}^4}{\kappa_{\lambda_2}^4} \frac{[1 + \delta_{\lambda_1}^2 \kappa_{\lambda_1}^2]^{3/2}}{[1 + \delta_{\lambda_2}^2 \kappa_{\lambda_2}^2]^{3/2}}
\]

\[
R = R_0^4 \left[ 1 + (2\pi)^2 \delta_{\lambda_2}^2 \frac{|\eta - \eta_0|^2}{\kappa_{\lambda_2}^2} \right]^{3/2}
\]

\[
(R/R_0^4)^{2\gamma} - 1 = (2\pi)^2 \delta_{\lambda_2}^2 \left( \frac{1}{\kappa_{\lambda_2}} - \frac{1}{\kappa_{\lambda_2}} \right)
\]

\[
\delta_{\lambda_2} = \sqrt{(R/R_0^4)^{2\gamma} - 1} \quad (4.3)
\]

Similarly, for a two-dimensional gaussian power spectrum (equation 2.21a):

\[
f_r (\theta_1, \theta_2; \lambda) = \kappa_0^4 F_0 \sigma^2 \delta^2 e^{-\kappa_0^2 \delta^2/2} \quad (A.2)
\]

\[
R = R_0^4 e^{-(\kappa_0^2 - \kappa_{\lambda}^2) \delta^2/2}
\]

\[
\delta_{g^2} = 2(\kappa_0^2 - \kappa_{\lambda}^2)^{-1} \ln(R/R_0^4)
\]

\[
\delta_{g} = [2\ln(R/R_0^4)]^{1/2} Q \quad (4.4)
\]

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