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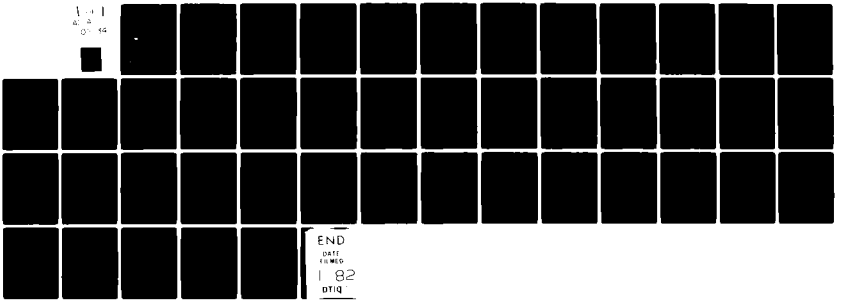

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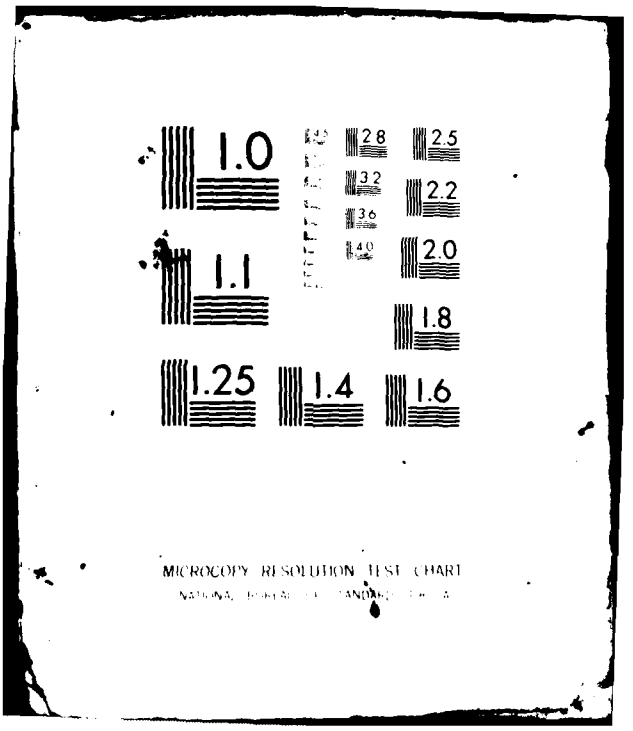
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SURFACE STATISTICS INVESTIGATIONS

Richard A. House II

October 1981

Final Report

Approved for public release; distribution unlimited.

AIR FORCE WEAPONS LABORATORY
Air Force Systems Command
Kirtland Air Force Base, NM 87117

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Results are reported of two investigations on surface structural properties of optical glasses. One project dealt with surface profiling analyses of a set of fused silica samples which had previously been laser damage tested. The other project had to do with the roughening of polished quartz and BK7 glasses by extended periods of ultrasonic cleaning.

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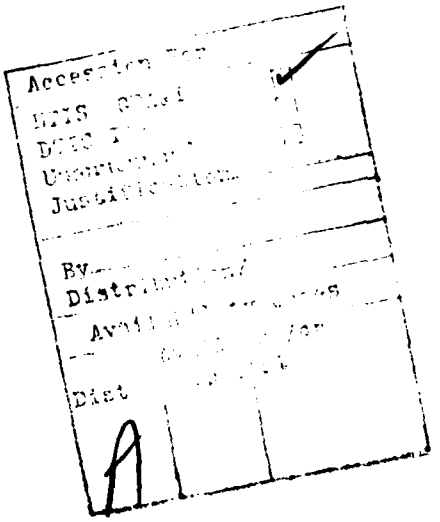
SUMMARY

This report presents the results of detailed surface statistical investigations carried out as two separate projects sponsored by the Air Force Weapons Laboratory. In the first project, a set of variously prepared and laser damage tested fused silica samples was subjected to surface profiling and total integrated scatter analyses. The samples had been damage tested in such a way that each retained a large section of virgin, untested surface area. The second investigation dealt with whether or not long periods of ultrasonic cleaning causes surface roughening and could, therefore, be a detrimental surface preparation operation. The basic conclusions were (1) that, although surface statistics vary greatly, laser damage threshold can be related to inferred surface roughness values, and (2) that ultrasonic cleaning of polished glass surfaces does not appear to be detrimental as long as the duration is kept to several minutes or less.



CONTENTS

I INTRODUCTION

High-power, Q-switched, solid-state lasers are limited in output intensity because of surface damage thresholds exhibited by optical elements. For many transparent materials of interest in the short-pulsed regime (less than 100 ns), surface thresholds are as much as a factor of 10 lower than corresponding bulk damage thresholds.

Extensive experimental observations previously showed that laser-induced breakdown at the surfaces of dielectrics is strongly correlated with inferred RMS surface roughness (Ref. 1). Becuase many different surface preparation techniques were used in making the test samples and yet the same basic functional relationship between threshold and roughness was observed, it was important to investigate the physical structure (i.e., surface statistics) of the samples. This report describes the results of that investigation.

The previous work demonstrated that the definite correlation between RMS surface roughness (as measured by any of several standard techniques) and pulsed laser damage thresholds in optical window materials (in the 40 ns pulse-width time domain) can be expressed by the following relationship:

$$E\sigma^m \sim K \qquad (1)$$

In Equation 1, E is the threshold optical electric field in V/cm), σ is the RMS surface roughness (in Angstorms), and m and K are material-dependent constants. The constant m was found to be approximately 0.5 for a wide range of finished silica window materials; the constant K, however, was found to vary as a function of particular finishing techniques for a given material. This parametric behavior led to the useful concept of a roughness-normalized threshold, which affords the opportunity for quantitatively comparing various surface preparation techniques and final finishes (Refs. 2 to 8).

A basic difficulty encountered in the cited research was a significant variation in quoted roughness values for any given sample. In general, the cited RMS value depended significantly on who measured the sample and what measuring technique was used. The three different techniques were: (1) Fringes of Equal Chromatic Order (FECO) - which uses interference fringes in

white light to optically contour surface height variations; (2) Total Integrated Scattering (TIS) - which infers roughness values from measurements of monochromatic light scattered by surface irregularities; and (3) Surface Profiling (SP) - which physically contours the surface height variations using a sharp mechanical probe (typical tip radius < 8 μm) in contact with and translated along the optical surface.

Except where they were unavailable, only FECO roughness values were used in the analyses of the previous work. This was done because it appeared that the FECO technique was the only one valid over the nearly two orders of magnitude range of roughness values investigated. Upon averaging the various roughness values for each subset of experimental samples and plotting the average values against the threshold values, it was found that the data the data still essentially fit relationship (1). However, it was clear that further surface characterization would be necessary to understand the differences among the standard surface roughness measuring techniques. Figure 1 (taken from Ref. 1) illustrates the nature of the difficulty, with respect to FECO and TIS roughness values. The data are for polished, uncoated fused silica. The open circles and dashed line are the FECO only data, and the filled circles and solid line are for the weighted averages of the FECO and TIS data. The Xs denote the positions of four of the tested samples based on the TIS roughness values determined for this report (Table 1).

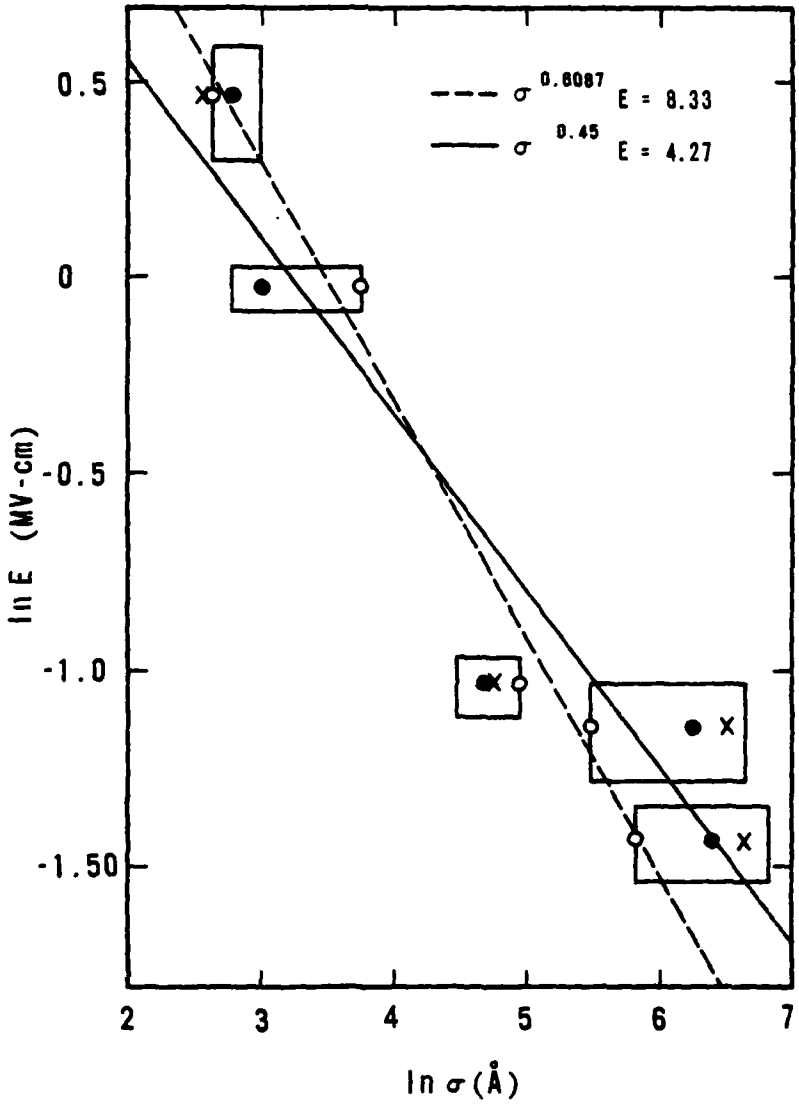


Figure 1. Threshold vs. Roughness, Conventional Samples. Horizontal spread: roughness measurement variation; vertical spread: threshold uncertainty. Open circles and dashed line: FECO only; filled circles and solid line: weighted average of FECO and TIS; Xs: positions of the four conventional samples of Table 1, based on the 5682 Å roughness values.

TABLE 1. ROUGHNESS MEASUREMENTS

Samples		NWC σ (TIS) (Å)	AFWL σ (FECO) (Å)	AFWL σ (TIS) (Å)	Δ32 percent	Δ42 percent
#	Type					
017	Conventional (Taly)	12.4	13.75	12.84	-10	-3
097	Conventional (Taly)	114.6	140	87.9	-18	30
093	Conventional (Taly)	668.8	243	372	175	80
085	Conventional (Taly)	757.1	335	348	126	118
088	Flame	74.3	82*	60.6**	--	23
058	Flame (Taly)	20.9	310*	13.32**	--	57
101	Flame	214.6	220*	56**	--	283
052	Etch	606.9	82	552.5	640	10
051	Etch (Taly)	216.1	243	234	-11	-8
055	Etch	725.4	325	534	123	36
008	S-Film (Taly)	9.3	13.75	--	-32	--
092	S-Film	504.4	243	--	108	--
103	S-Film	432.5	335	--	29	--
042	M-Film	61.4	13.75	--	347	--
084	M-Film	200.0	82	--	144	--
095	M-Film	416.8	220	--	89	--

TABLE 1. ROUGHNESS MEASUREMENTS (concluded)

Samples		NWC σ (TIS) 5682 Å	AFWL σ (FECO)	AFWL σ (TIS) 6328 Å	Δ32 percent	Δ42 percent
#	Type					
062	Subsurface (5μ)	19.3	12.8	10.1	51	91
063	Subsurface (5μ)	19.4	12.8	10.1	52	92
065	Subsurface (12μ)	13.5	8.26	9.15	63	48
066	Subsurface (12μ)	10.6	8.26	9.15	28	16
071	Subsurface (20μ)	12.4	11.61	10.9	7	14
073	Subsurface (30μ)	22.2	16.7	8.87	33	190
074	Subsurface (30μ)	20.6	16.7	8.87	23	132
SH12-2	H-Ion	21.0	--	14	--	50
SA12-1	A-Ion	26.0	--	31	--	-16
SX12-2	X-Ion (Taly)	16.5	--	36	--	-54
SP1-2	Bowl-Feed (Taly)	24.8	--	13	--	91
064	U-Sub. (5μ)	13.4	12.8	10.1	5	33
068	U-Sub. (12μ)	15.0	8.26	9.15	82	64
076	U-Sub. (30μ)	16.8	16.7	8.87	1	89

(Taly) denotes Talystep surface profile analysis at NWC
* before flame polishing
** after flame polishing

II ROUGHNESS INVESTIGATIONS

Thirty samples of Optosil I fused silica that had previously been treated by various finishing techniques and subjected to laser damage testing were submitted to the Michelson Laboratory, Naval Weapons Center, China Lake, California for surface topography characterization. Although prior sample handling has been detailed in Reference 1, a brief summary is presented below.

BACKGROUND

Surface Preparation

Nine groups of samples were prepared for investigation. These groups differed basically in surface topography, including a roughness variation.

a. *Conventional:* Complete controlled grinding plus standard polishing using barnesite.

b. Flame: Conventional plus flame polishing.

c. Etch: Conventional plus nitric acid etching.

d. S-Film: Conventional plus overcoating with half-wave thickness of SiO_2.

e. M-Film: Conventional plus overcoating with half-wave thickness of SiO_2.

f. Subsurface: Incomplete controlled grinding plus standard polishing using barnesite.

g. Ion: Complete controlled grinding plus standard polishing using jeweler's rouge plus ion polishing.

h. Bowl Feed: Complete controlled grinding plus superpolishing using jeweler's rouge.

i. U-Sub: Preparation f. followed by ultrasonic cleaning for 90 minutes.

Prior Characterization

The samples were characterized prior to damage testing either by FECO or by TIS, or by both. The FECO measurements were made on representative members of each specific batch of samples rather than on every final sample. The TIS measurements (at 6328 Å) were made on all final samples, except

those with a dielectric overcoat, by scattering the light off a central metal film disk (a 1/2 inch diameter coating or Mo of Al deposited on the sample). Prior to irradiation of any specific sample site, the site was inspected by a HeNe laser beam to ascertain that it was free of scattering centers.

Damage Testing

These samples were laser damage tested in such a way that a damage event at one site on a given sample did not contaminate any other site on the sample. Laser beam parameters were the following: 1.06 μm wavelength, 40 ns pulse width, TEMoo mode structure, and 147 μm diameter spot size (at the e^{-2} power points). Each sample site was irradiated only once, and various indicators were employed to determine that a damage event did or did not occur.

MEASUREMENTS AND RESULTS: I

Characterization and damage testing of the samples were completed by December, 1975, and the China Lake surface topography characterization was begun in December, 1976. In the intervening period, the samples were stored in their closed containers in a cabinet. Occasionally, they were opened for purposes of visual inspection and to select samples for the surface topography characterization. This time sequencing apparently has a bearing on the results obtained.

Characterization at China Lake in 1976 was done by measuring the TIS surface roughness of each sample at 5682 Å. Samples were not cleaned other than blowing them with nitrogen gas. Table 1 lists the samples and the results of these and previous measurements. All roughness values are in Angstromes. The first column designates the type of sample examined, the second column gives the 1976 TIS roughness values, the third column shows the 1976 vendor-supplied FECO results, and the fourth column lists the 1976 TIS results at 6328 Å, also supplied by the vendor. The fifth and sixth columns present the percent change from the 1975 values. For instance, a column five value is the number

$$\Delta_{32} = 100\ \frac{\sigma_{NWC}(TIS - \sigma_{AFWL}(FECO)}{\sigma_{AFWL}(FECO)} \qquad (2)$$

Column six is similarly constructed, using σ_{AFWL} (TIS) instead of σ_{AFWL} (FECO).

Immediately apparent from the table is the result that the final roughness vaues are greater than the initial roughness values in more than 80 percent of the cases and that individual increases are much greater than individual decreases. In fact, the decreases can be essentially discounted because their magnitudes are smaller than the typical consistency of measurements made by different operators of different instruments.

The indicated wholesale increase in inferred surface roughness values led to a closer examination of the sample surfaces. The essential result is that the samples were found to have large numbers of tenaciously held particulates on their surfaces. Moreover, the particulate matter was, in many cases, not removable using dry nitrogen and an electron gun. This could indicate a number of things, such as material migrating out of the sample or foreign material having deposited and become partially or completely bound to active surface sites. Presumably, such site activiation could be a result of prior high energy laser irradiation. However, the effect was just as noticeable on the central, unirradiated regions of the samples as well as on the peripheral regions. What has caused this is not known at present; but examination of the data shows that the basic threshold verses roughness relationship still holds. That is, the value of m is still approximately 0.5 although the constant K has changed for each individual surface treatment.

To verify and quantify the observation concerning particulate matter on the surfaces, two samples were selected for detailed analysis using the Michelson Laboratory's recently developed computerized SP capability. The following four paragraphs are taken, essentially verbatim, from a China Lake letter report on this effort.

Sample #017

The smoothest sample of the conventional series (smoothest by virtue of the lowest NWC TIS roughness value) was chosen for statistical analysis using the Talystep surface profiling instrument. The standard diamond tip (8 μm radius) was used with a 2 mg loading. Previous experiments have shown

that this loading does not mark the molybdenum films on the selected sample. Five scans were made on different portions of the molybdenum-coated surface. Each scan was 1.037 mm (1,037 μm) long and data were taken at 0.061 μm intervals. Thus, there were 17,000 data points per scan to use in the statistical analysis. The average height and slope distribution functions for the five scans are shown in Figure 2. In all cases, the surface was extremely smooth (~ 2 Å RMS); but it was covered with isolated bumps which dominated the surface statistics, making the height distribution function skewed. Since there were no holes in the surface, the mean surface level (calculated by putting a least squares straight line through all the data points) was slightly above the smooth surface, producing the skewed effect. The bumps dominated the height statistics, making the RMS height much larger than it would have been if only the smooth part of the surface had been present. Thus, the Gaussian height curve, which has the same area under it as the measured histogram, does not nearly fit the measured histogram. The bumps on the surface also dominate the slope distribution function. The steep slopes are at the edges of the bumps and distort the histogram, making the measured RMS slope much larger than for the surface without the bumps. Figure 3 shows similar results for the smoothest of the flame polished series.

Since there were so many data points (17,000) for the 1.037 mm scan length, it took too much computer time to calculate the autocovariance function for the entire scan length. Therefore, some compromises were made. The initial portion of the autocovariance function was calculated for the first 1,000 points (i.e., a lag length of 61 μm) to show the structure of the initial spike. This calculation took one hour on an HP 2100 minicomputer. A typical example for the smoothest conventional sample is shown in Figure 4. In this figure, the dotted curve is the initial portion of the solid curve, except that the horizontal scale has been expanded by a factor of five. Also shown are computed values of the autocovariance function $G(\tau)$ for increasing separation of pairs of data points (in units of $\tau_o = 0.061\mu m$). Next all 17,000 data points were averaged in pairs (i.e., $\tau_o = 0.122\mu m$) and the autocovariance function was calculated for the first 4,000 resulting "points" (lag length of 488 μm). This calculation took about three hours on the minicomputer. A typical example is shown in Figure 5 along with the

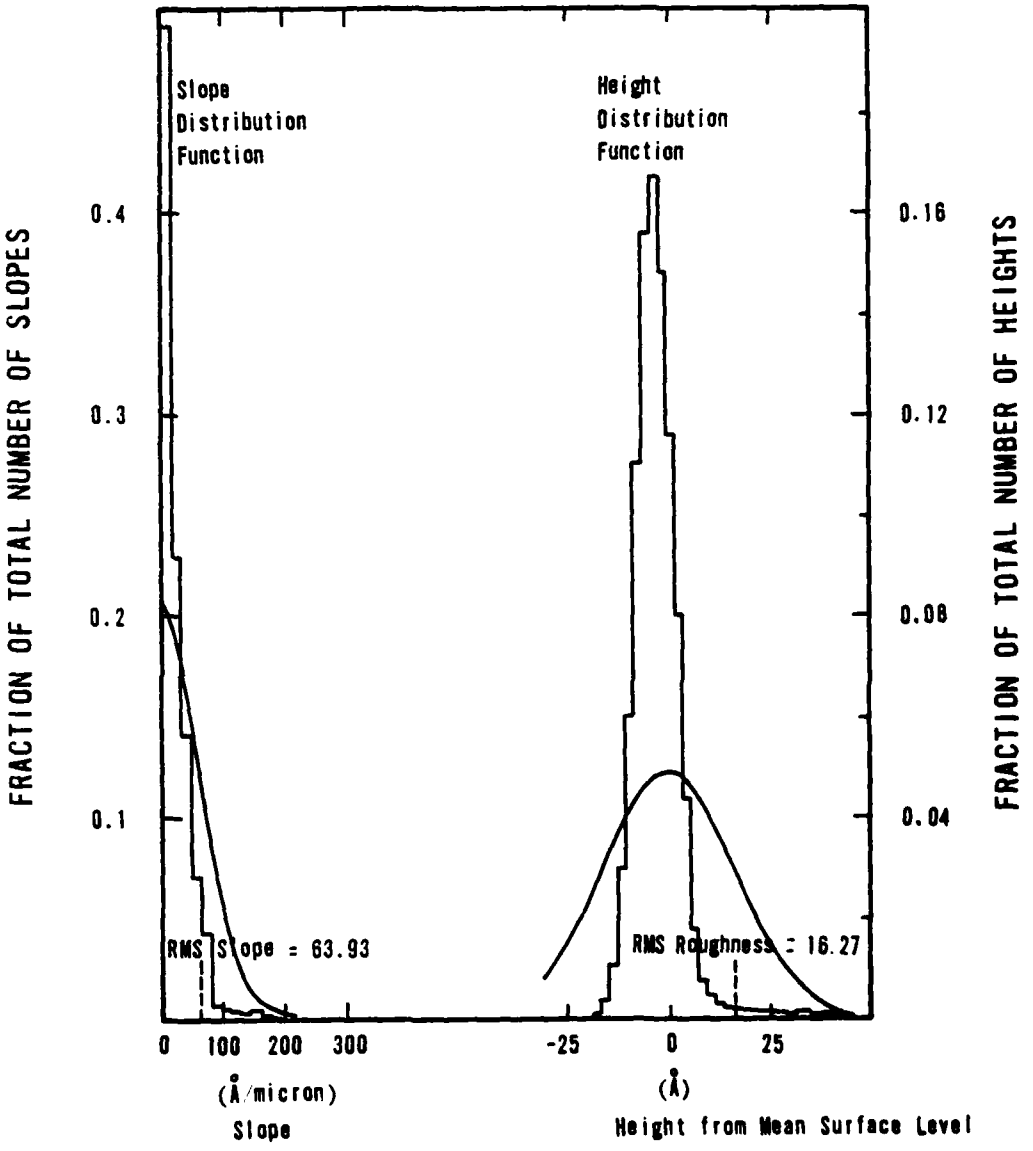


Figure 2. Surface Distribution Functions, Sample #017. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

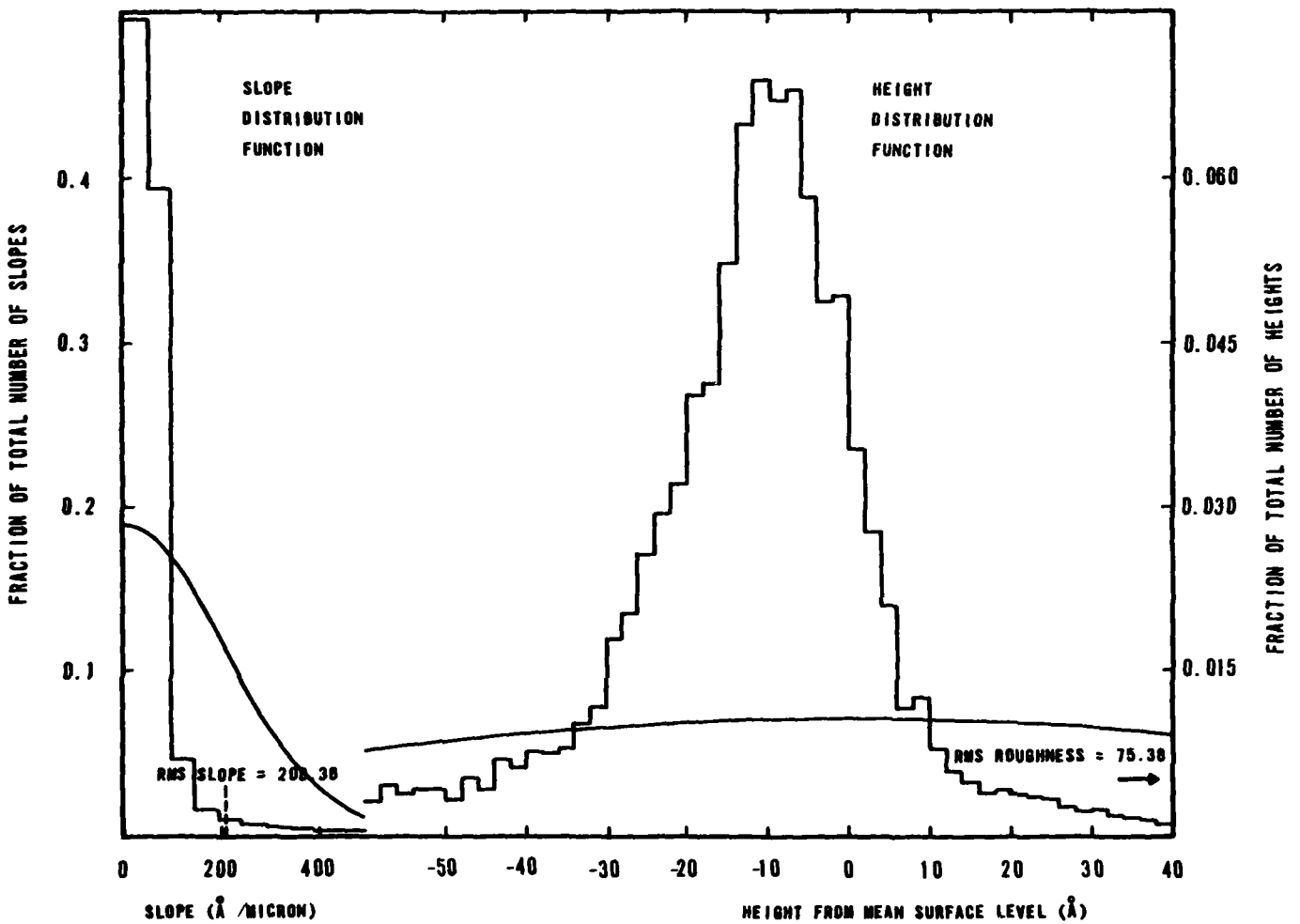


Figure 3. Surface Distribution Functions, Sample #058. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

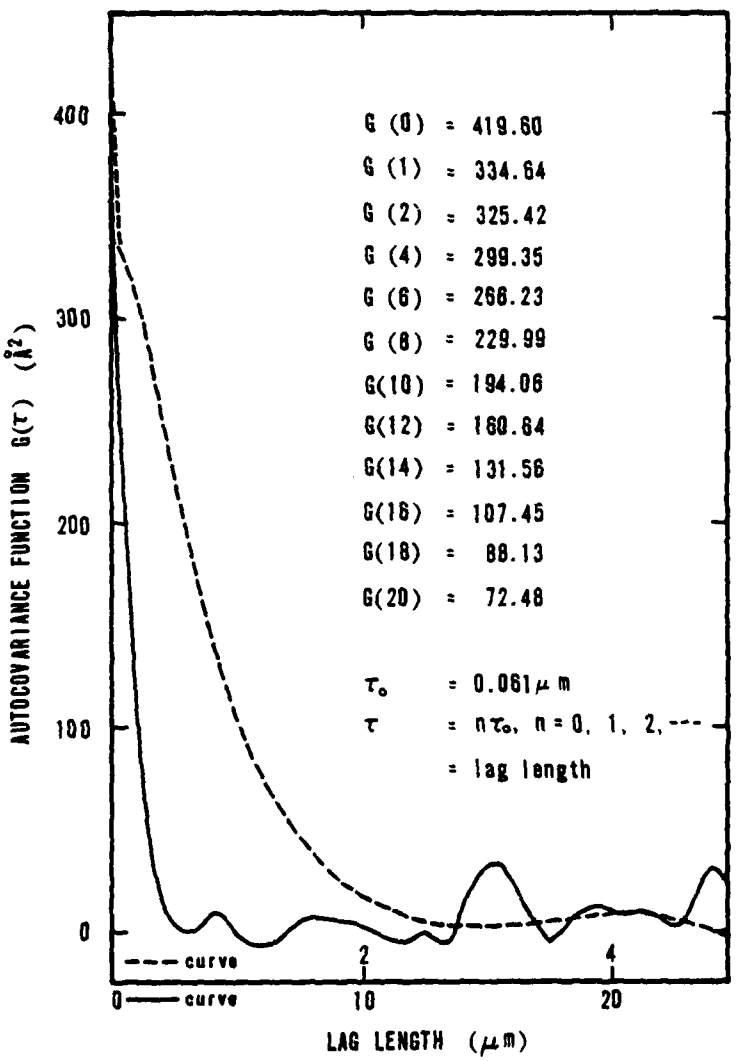


Figure 4. Autocovariance Functions, Sample #017. Initial portion of function, shown at two magnifications. Data base: first 1000 Talystep points and minimum interval (i.e., τ_o = 0.061 μm).

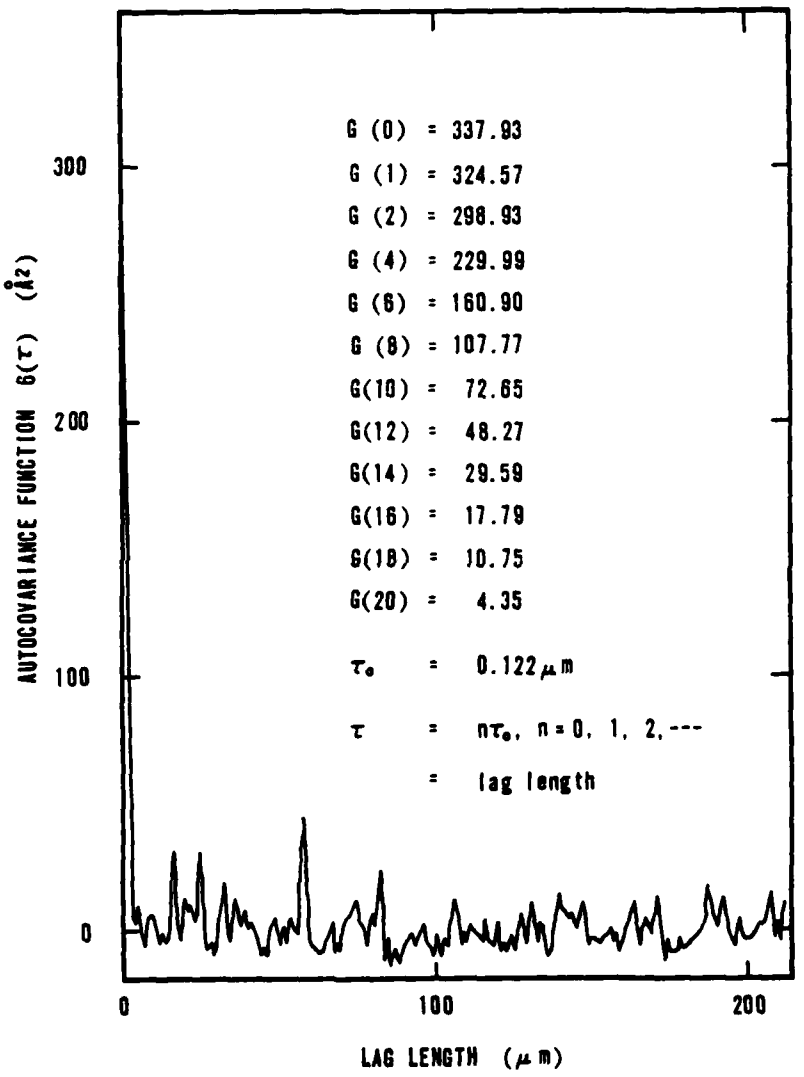


Figure 5. Autocovariance Function, Sample #017. Initial portion of function, based on averaged data. Data base: first 4000 values after averaging the 170000 raw data values in pairs. Interval is twice minimum (i.e., τ_o = 0.122μm).

specific values of G (τ). Finally, the original data points were averaged in blocks of 32, making the basic increment, τ_o, equal to 1.952 μm. This increment is nearly equal to the basic increment used in the China Lake FECO analyses. Hence, the lag length was 1,036 μm, and a plot of the data (with specific values of G (τ included) is shown in Figure 6. All these autocovariance functions show that (1) the correlation is very short ranged (less than 2.5 μm) and is mainly caused by the bumps (Thus, the correlation lenght can be considered to be a crude measure of the bump width); (2) the remainder of the correlation is negligible compared to the correlation produced by the bumps, so that there are essentially no long range correlation effects; and (3) if the original surface height data are averaged in pairs or in groups of 32 ("FECO data"), the values of G(o) are smaller than the unaveraged value. Hence, the RMS roughness value calculated from averaged surface data will be smaller. (The RMS roughness value is the square root of G(o)).

Since the dominant feature on the sample of Figure 2 and on the other samples are isolated particulates, a method was devised for obtaining statistics of these particulates: (1) A 610 μm length of the surface was scanned with the Talystep and 10,000 data points were taken with a spacing of 0.061μm. (2) A least squares straight line was put through all the data to obtain a mean surface level. (3) All points (from the bumps) whose deviations from the least squares straight line that were more than four times the average deviation were eliminated. (Steps (2) and (3) were repeated until either there were no points whose deviations were more than four times the average deviation, or the iterations had been repeated ten times.) (4) Now, there was essentially a least squares straight line through the points on the surface where there were no bumps. This line served as a reference level for determining the heights of bumps. (5) Using the original surface data, the deviation of each point from the reference level was calculated. When this deviation was more than four times the average deviation of the points without the bumps, a bump was defined to have occurred. The width of the bump and its maximum height above the reference level were calculated. (6) The data in (5) were printed and punched on paper tape. Then the average number of bumps per scan was calculated as was the fractional coverage of

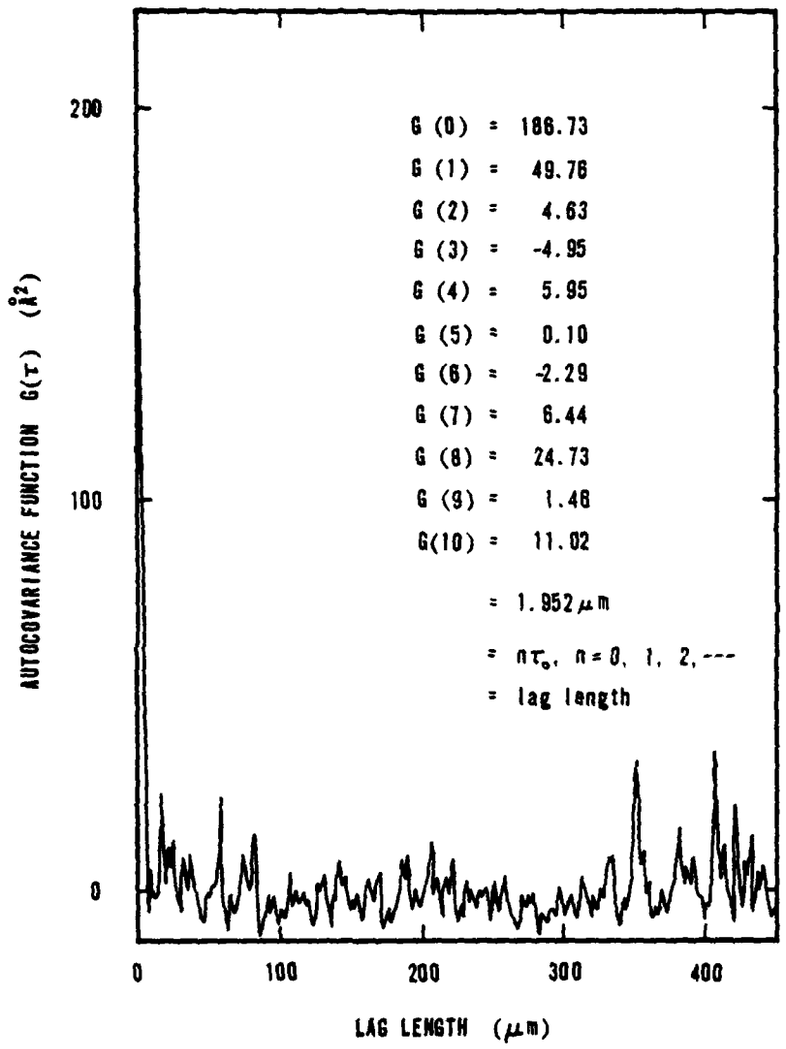


Figure 6. Autocovariance Function, Sample #017. Initial portion of function, based on averaged data: simulation of FECO data. Data base: 17000 raw data values averaged in blocks of 32 (i.e., τ_o = 1.952μm).

bumps (i.e., the total length of bumps in a scan divided by the total scan length--610 m). Also, the average height distribution function for all the bumps in all scans was calculated and plotted. For the smoothest conventional sample (Figure 2), there was an average of 31.75 particles/scan and the fractional coverage was 0.0422. The particle height distribution function (Figure 7) shows that a majority of the particles had heights in the 20 to 30 Å range above the reference level. The largest particle had a height of 205 Å.

Sample #058

The particle analysis was also performed on the smoothest flame polished sample (Figure 3). There was an average of 56.00 particles/scan and the fractional coverage was 0.0718. The particle height distribution function (Figure 8) shows that a majority of the particles had heights in the 30 to 50 Å range above the reference level. The largest particle measured had a height of 906 Å. Note that the particle height distribution function (Figure 8) and the surface height and slope distribution functions (Figure 3) are as distinctly non-Gaussian as the corresponding functions (Figures 7 and 2) for the smoothest conventional sample. Autocovariance functions were not calculated for this surface, but they would have been almost identical to the ones determined for the conventional sample (Figures 4, 5, and 6).

Table 2 summarizes the surface statistical information on the above two samples. Column three gives the Talystep RMS roughness values of the surfaces - including the effect of the particles. Column four gives the RMS surface slopes in Å/micron and in degrees. By subtracting the particle data from the overall data, the roughness values of the background surfaces can be obtained. The background RMS roughness values are given in column five. The subtracted data represent the particles only, and their RMS height values are given in column six. For comparison purposes, the TIS RMS roughness values (including the effect of the particles) are given in column seven and are the same values found in Table 1.

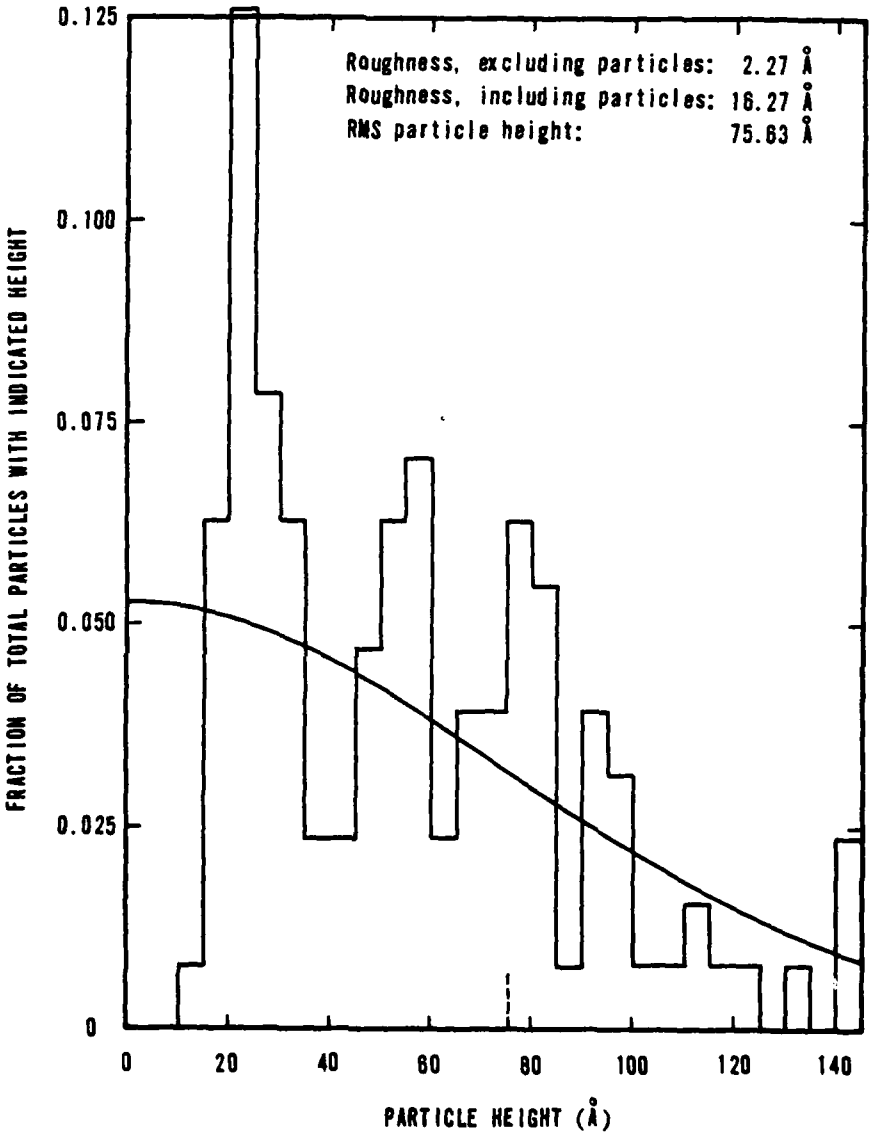


Figure 7. Particle Height Distribution Function, Sample #017. Smallest particles counted have heights greater than four times the average deviation of the background surface.

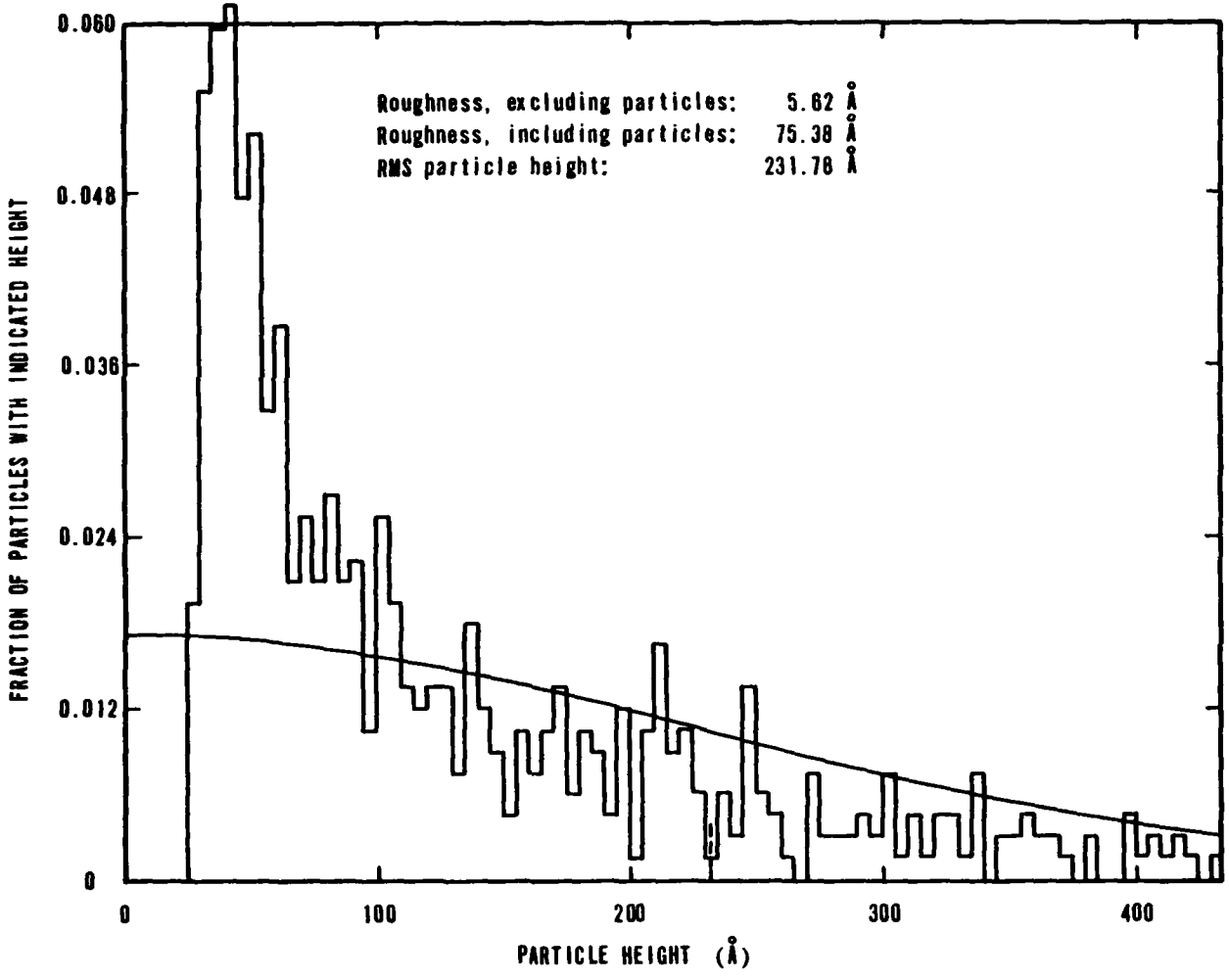


Figure 8. Particle Height Distribution Function, Sample #058. Smallest particles counted have heights greater than four times the average deviation of the background surface.

TABLE 2. SURFACE STATISTICS OF TWO SAMPLES

Sample #	Sample Type	σ (Taly) (Å)	slope (Taly) (Å/micron)	σ_b (Taly) (Å)	h_p (Taly) (Å)	σ (TIS) (Å)
017	Conventional	16.27	$63.93 = 0.37^o$	2.27	75.63	12.4
058	Flame	75.38	$208.36 = 1.19^o$	5.62	231.78	20.9

MEASUREMENTS AND RESULTS: II

1976 Data

Similar Talystep measurements were also made for the smoothest Etch (#051), S-Film (#008), X-Ion (#SX12-2) and Bowl Feed (#SPI-2) samples. As in the previous cases, these samples also contained significant amounts of particulate matter which generally were removable using mild soap and water. The basic results of these measurements (some of which were previously published in Ref. 7) are the following:

1. The Etch sample was very rough, and its surface statistics were dominated by deep holes in the surface. Hole depths generally ranged from 250 to 1000 Å, but several holes as deep and as wide as 1-2 μm were also detected. The presence of many deep holes caused the mean surface level to shift below the relatively-smooth background surface of the sample. As a result, the height distribution function (Figure 9) is skewed to the right and distinctly non-Gaussian. In addition, the presence of very large slopes on the walls of the holes caused the slope distribution function (also Figure 9) to extend far to the right and be severely non-Gaussian.

2. The S-Film sample, except for relatively few unremoved particles (which dominated the inferred TIS roughness values shown in Table 1), was exceptionally smooth (i.e., less than 3 Å) over much of its surface. The height and slope distribution functions (Figure 10) were both Gaussian.

3. The X-Ion sample exhibited a fairly smooth surface by TIS measurement (i.e., the value shown in Table 1) but a fairly rough surface by SP measurement (about 60 Å). There is the possibility that the bumps and holes on the surface were spaced closely enough together that the TIS wavelength of 5682 Å was not completely resolving them. However, there are too few available samples to positively state that this is the true explanation of the measurments. The height and slope distribution functions (Figure 11) were both reasonably Gaussian.

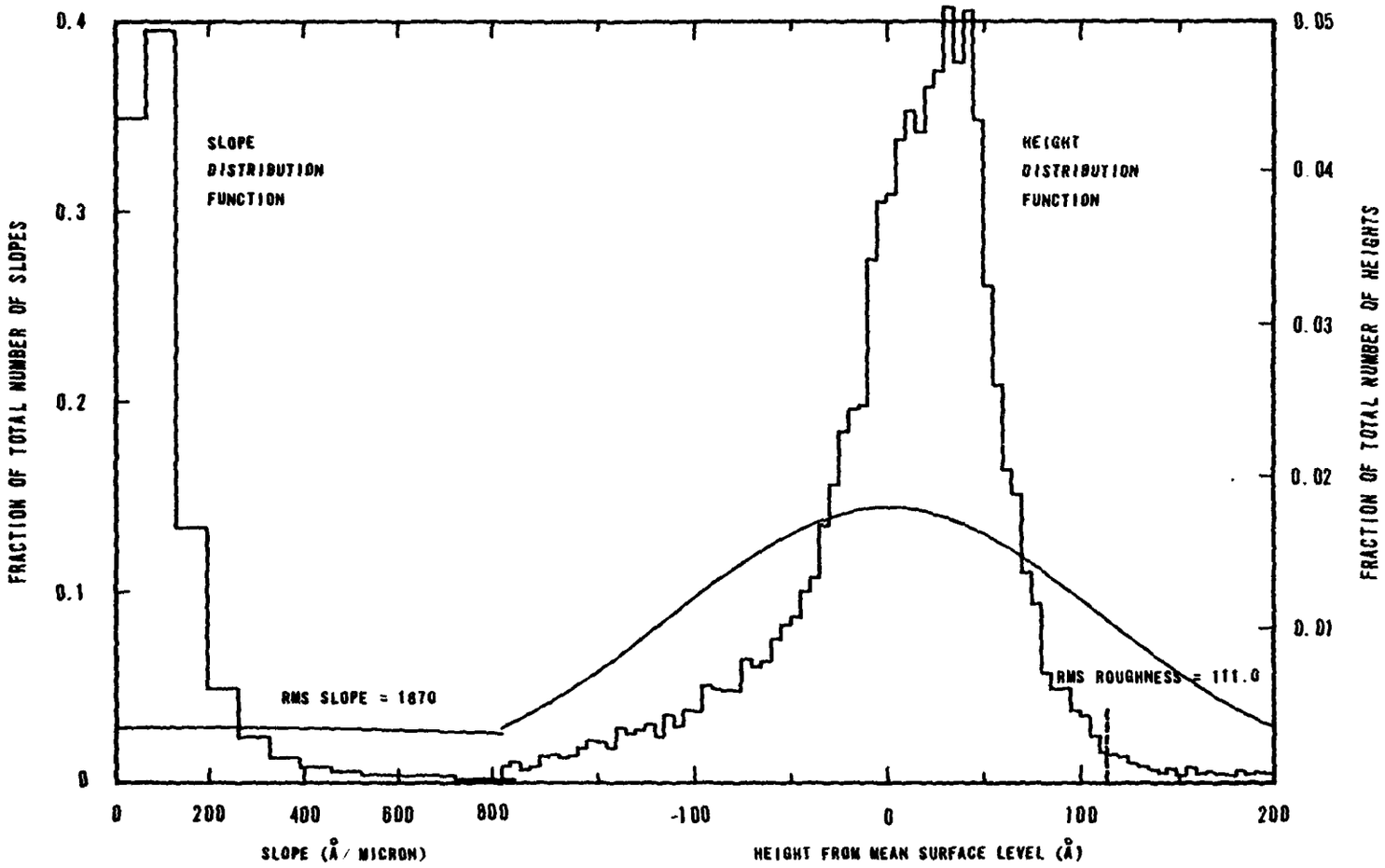


Figure 9. Surface Distribution Functions, Sample #501. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

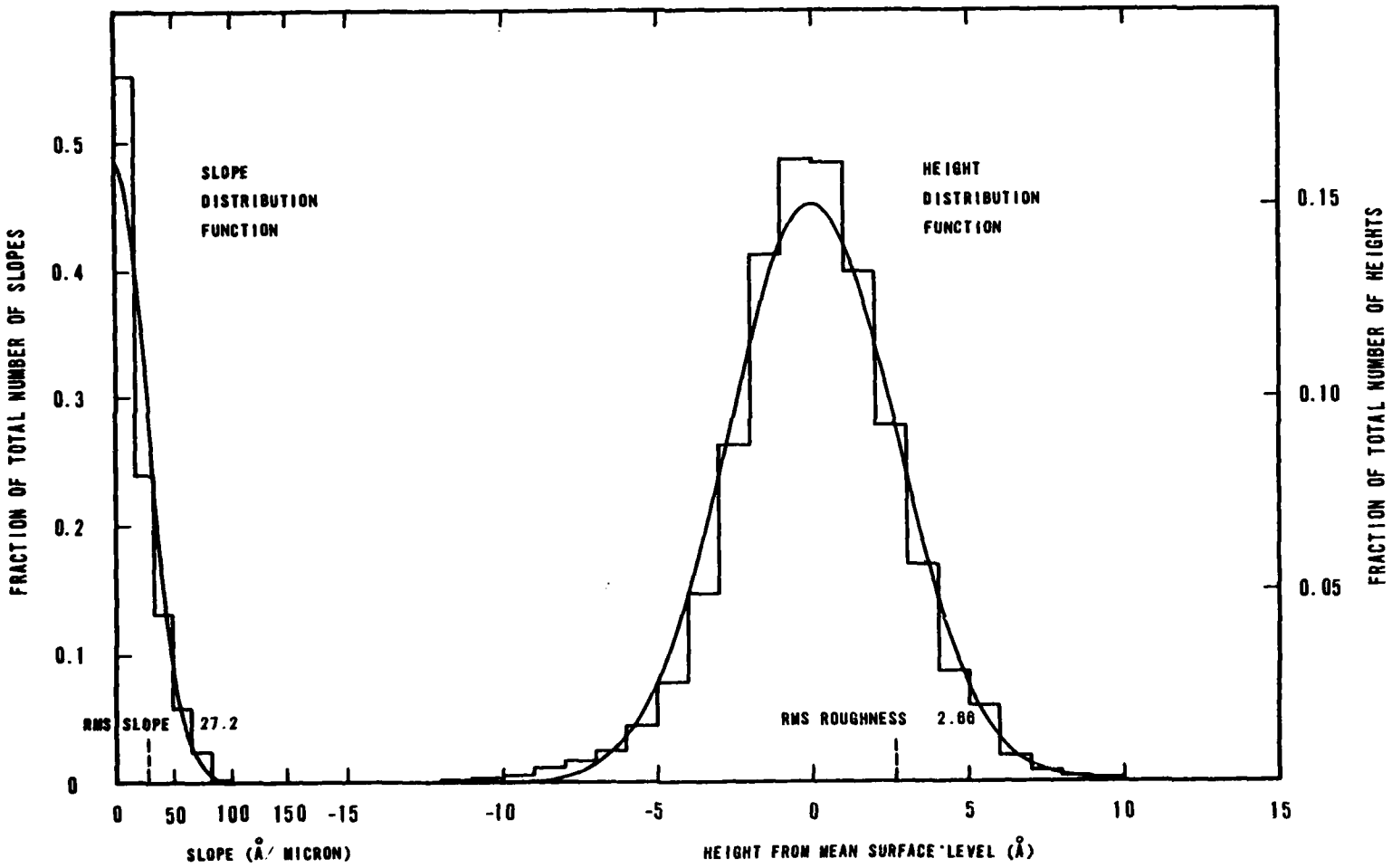


Figure 10. Surface Distribution Functions, Sample #008. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

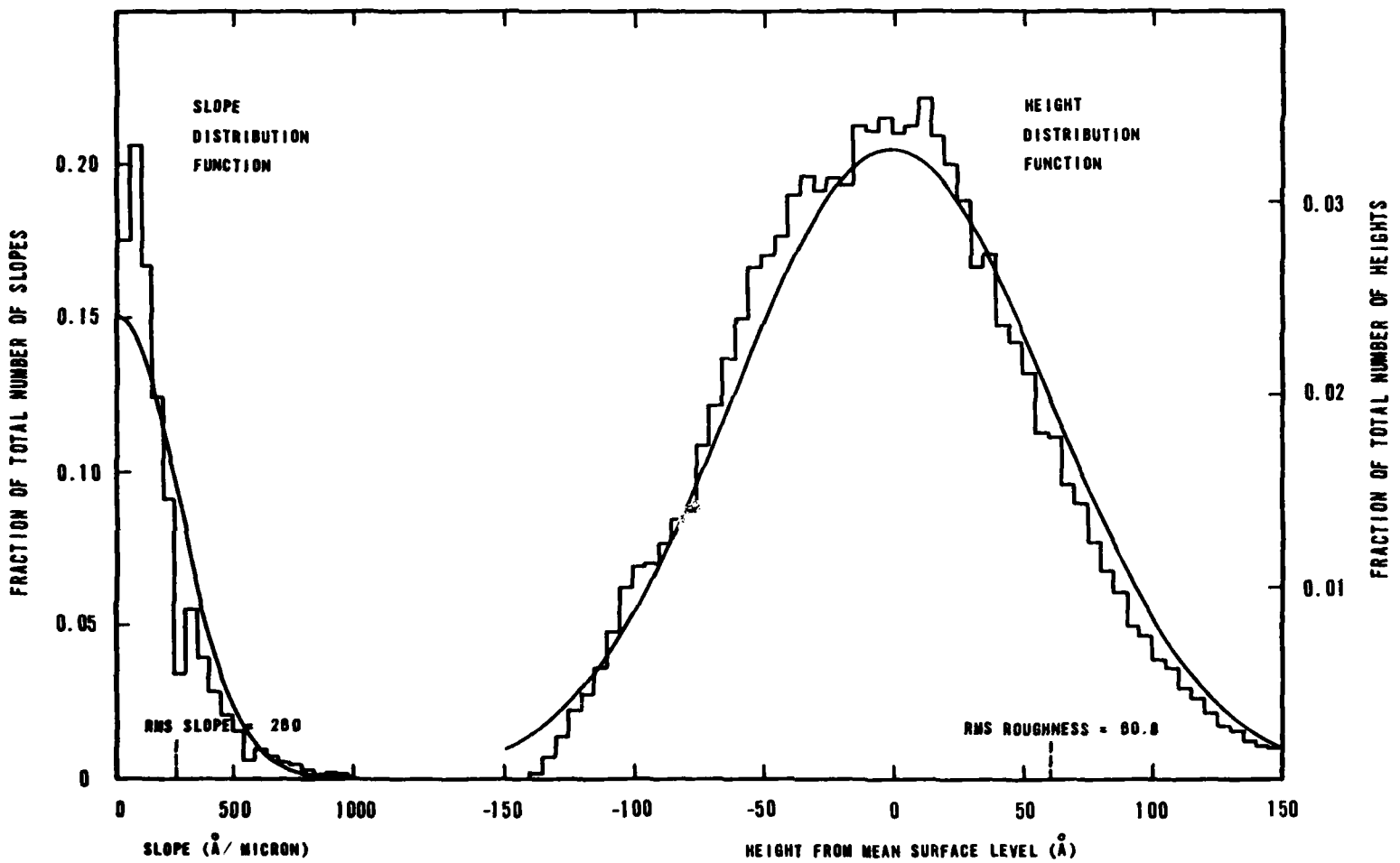


Figure 11. Surface Distribution Functions, Sample #SX12-2. Histograms: Talystep data; GAUSSIANS: fitted assuming identical areas and RMS values.

4. The Bowl-Fee sample surface scan looked like a fresh-feed sample surface scan in that it showed a lot of structure. There were also many particles having heights in the 100-500 Å range, and these could not be removed by two careful cleanings. This sample had a non-Gaussian height distribution although the slope distribution was nearly Gaussian. Figure 12 shows both the distributions. The presence of particles, for reasons similar to those stated for the Etch sample, caused the height distribution to skew to the left. That distribution is therefore not Gaussian. On the other hand, the initial portion of the autocovariance function (not reproduced in this report) had a 1/e width of about 0.8 μm. If we take this as a measure of the width of the surface "bumps" or particles, then most of the surface slopes are small and we expect the distribution of slopes to be nearly Gaussian.

Table 3 summarizes the surface statistical information on the above four samples. It is constructed similarly to Table 2 excet that the background and particle statistics were not explicitly separated.

1978 Data (Four Samples)

Althouh not originally intended, detailed surface scans of the conventinal samples became necessary because of the interesting and varied results being obtained on the other samples. These analyses were funded separately, however, and occurred later in time. Because of scheduling requirements, the data were not available until 1978.

Table 4 presents the essential results for samples 016, 097, 093, and 085. Column three gives roughness data measured in 1975 before the samples were damage tested. Column four repeats Table 1, except for sample 016. The last two columns give the Talystep surface analysis results. Sample 016 is a duplicate of sample 017 - they were polished in the same block - and both were damage tested.

Comments similar to those made previously apply to these four samples. None of them exhibited Gaussian behavior in either the height or slope distributions. This fact is one reason why TIS roughness values sometimes disagree markedly from Talystep or FECO roughness values. The inference of a TIS roughness relies on the use of a formalism which requires (a)

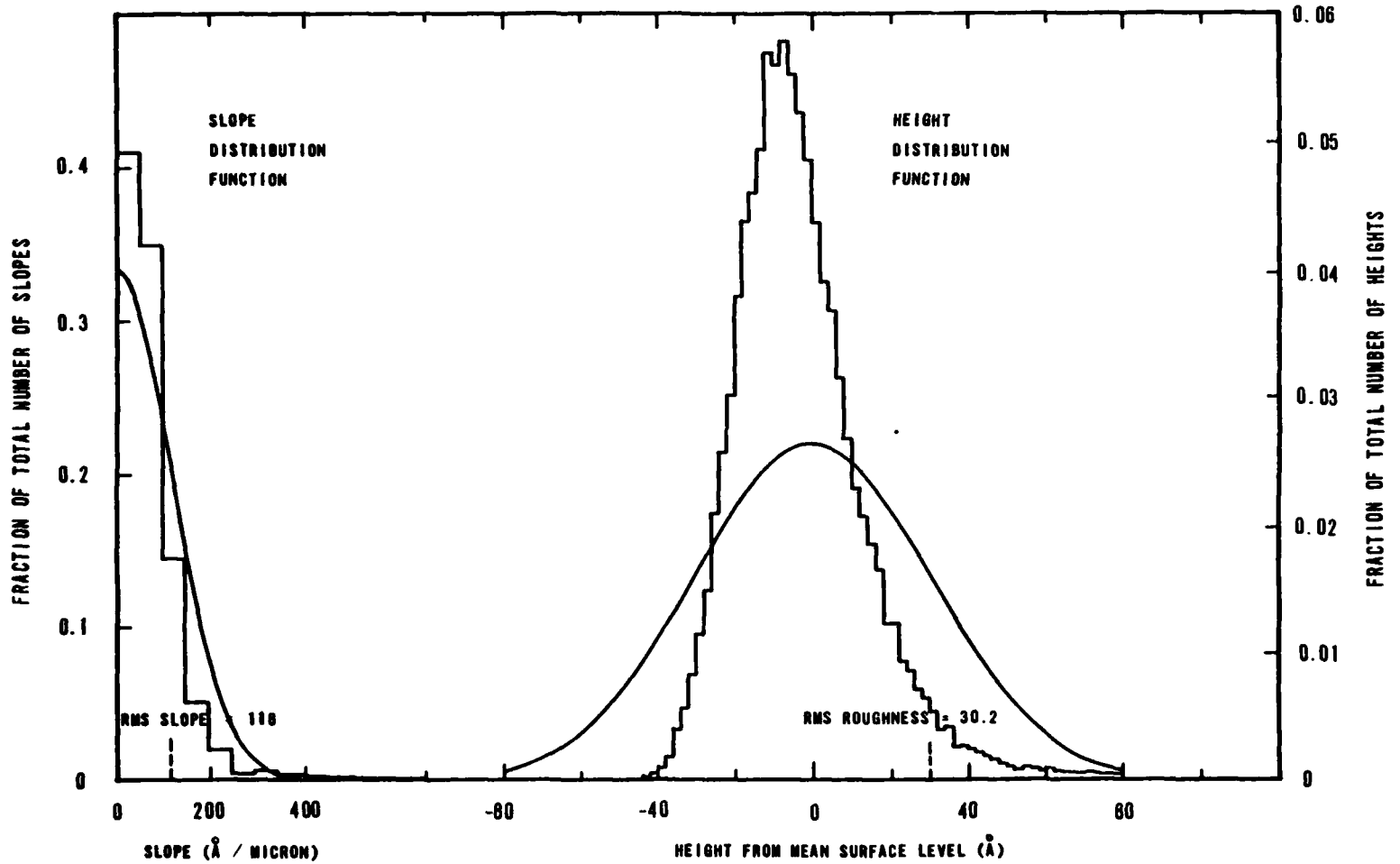


Figure 12. Surface Distribution Functions, Sample #SPI-2. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

TABLE 3. SURFACE STATISTICS OF FOUR SAMPLES

Sample #	Type	σ (Taly) (Å)	slope (Taly) (Å/micron)	σ (TIS) (Å)
051	Etch	111.0	1870 = 10.6°	216.1
008	S-Film	2.66	27.2 = 0.16°	9.3
SX12-2	X-Ion	60.8	260 = 1.49°	16.5
SPI-2	Bowl-Feed	30.2	118 = 0.67°	

TABLE 4. SURFACE STATISTICS OF FOUR SAMPLES

Sample #	Type	1975 σ_o (TIS) (Å)	1976 σ_o (TIS) (Å)	1978 σ_o (Taly) (Å)	1978 slope (Taly) (Å/micron)
016	Conventional	15.6	--	14.3	68.22 = 0.39°
097	Conventional	99.8	114.6	180	420 = 2.41°
093	Conventional	561.8	668.8	592	787 = 4.50°
085	Conventional	632.7	757.1	738	801 = 5.09°

that the roughness be much, much smaller than the wavelength of the light used to make the measurement and (b) that the heigh distribution be Gaussian.

As can be seen from the table and from Figures 13-16, the four conventional samples exhibit characteristics similar to those described earlier. The two smoother samples (016 and 097) appear to be dominated by particulate matter, since the height distribution is skewed left. The two very rough samples are skewed right, suggestive of dominance by holes and/or scratches. Except in the case of sample 016, the particle height data were not separated from the overall data. For sample 016, the background surface was determined to have a roughness of 4.3 Å and the RMS particle height was found to be 70.8 Å.

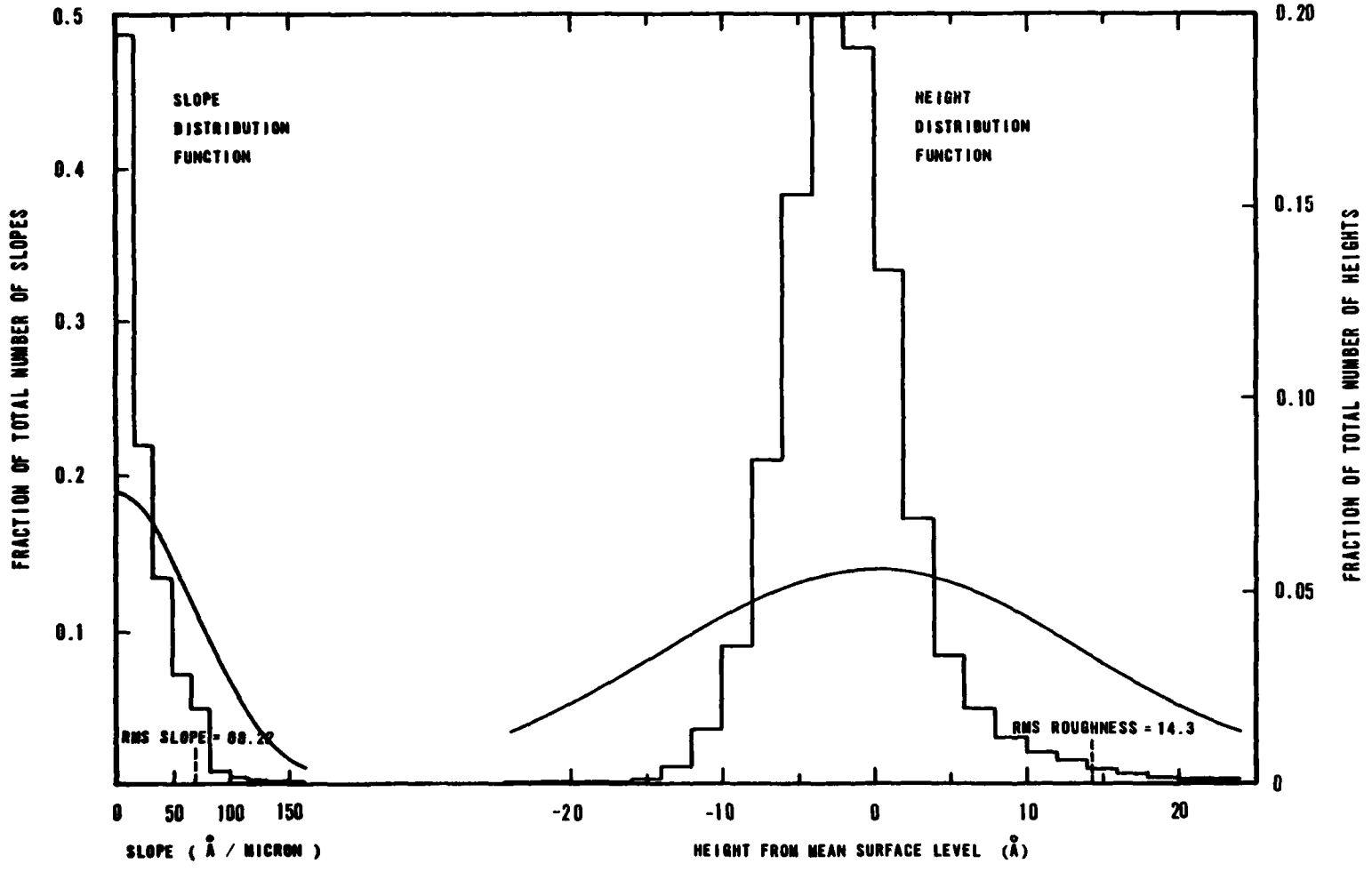


Figure 13. Surface Distribution Functions, Sample #016. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

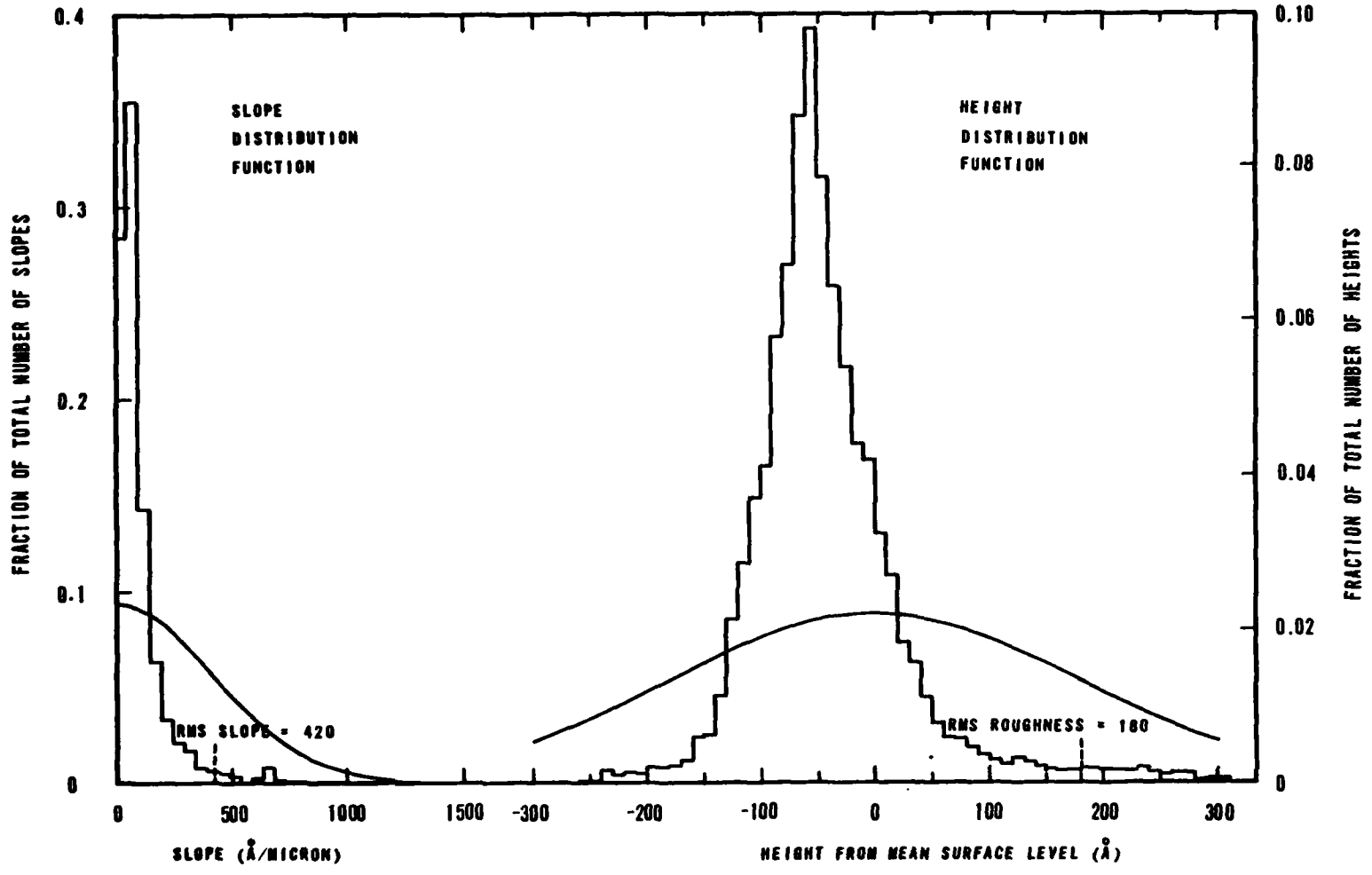


Figure 14. Surface Distribution Functions, Sample #097. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

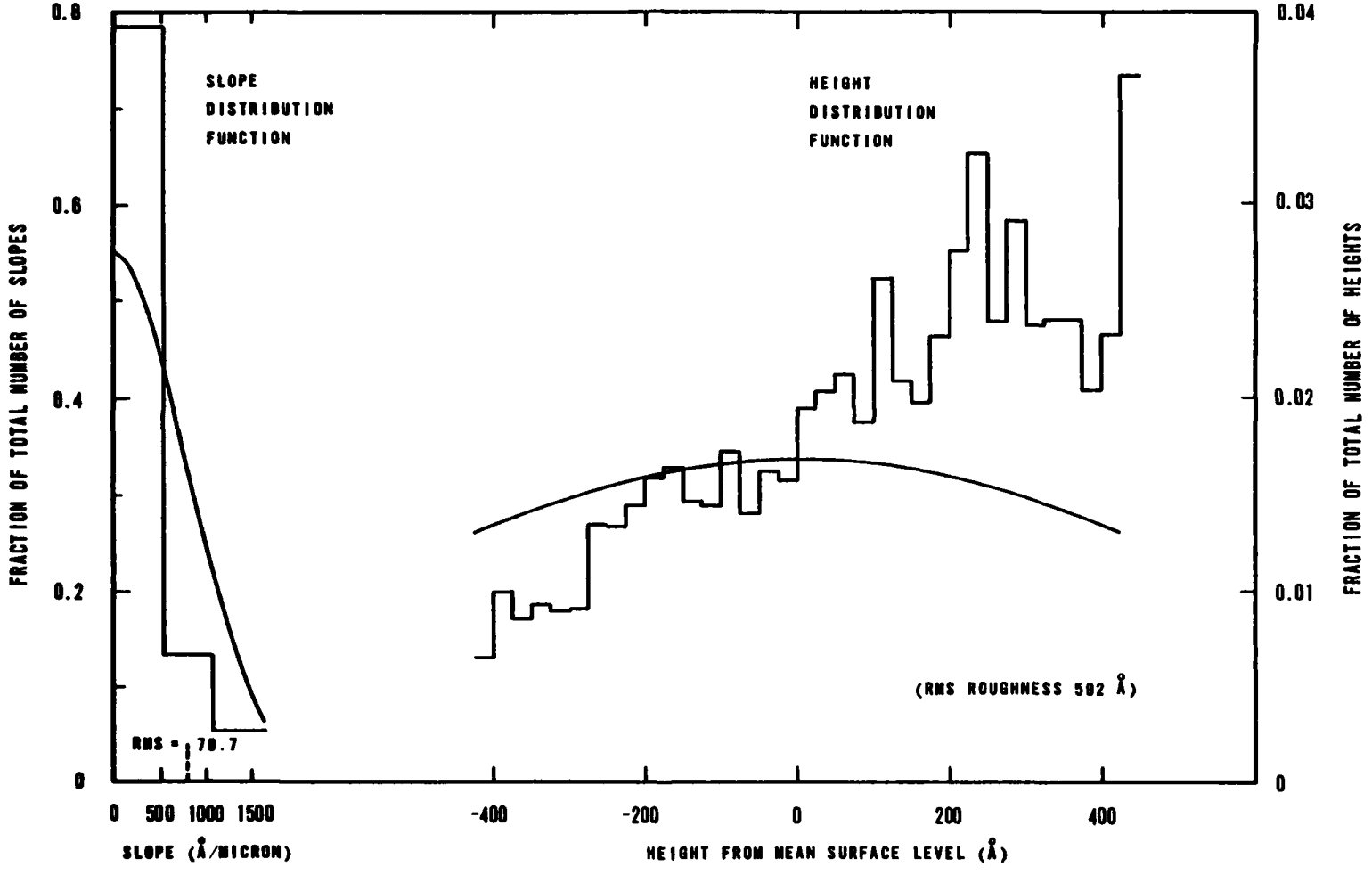


Figure 15. Surface Distribution Functions, Sample #093. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

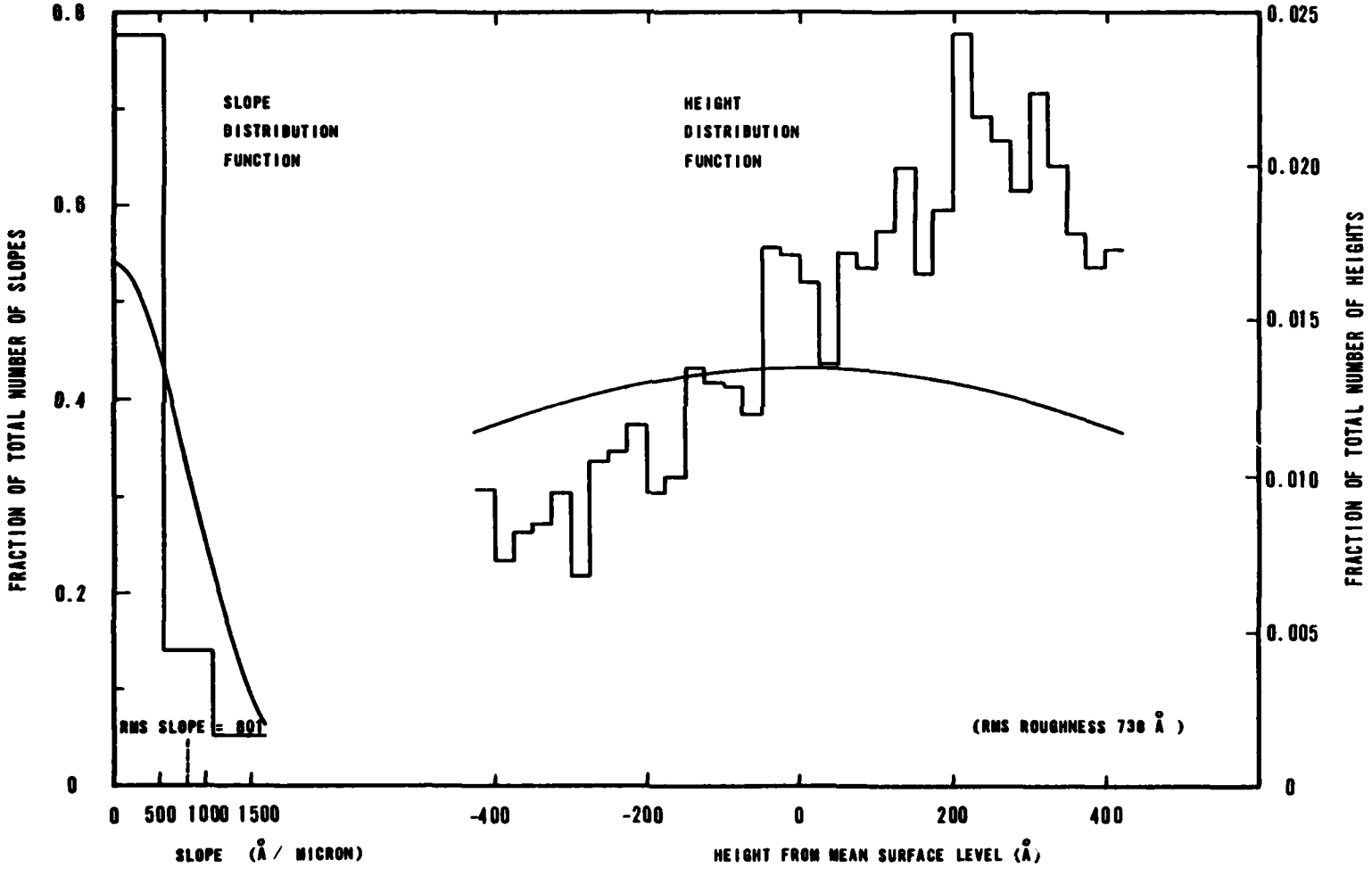


Figure 16. Surface Distribution Functions, Sample #085. Histograms: Talystep data; Gaussians: fitted assuming identical areas and RMS values.

III ULTRASONIC CLEANING INVESTIGATION

BACKGROUND

Although it is not usually considered a surface preparation technique in the sense used in the previous section, ultrasonic cleaning may have significant effects on surface structure and on laser damage threshold. In fact, the work described in Reference 2 deals with the observations at the Air Force Weapons Laboratory and elsewhere that long periods of ultrasonic cleaning increased the surface roughness of glasses and metals. The effects of such increases on laser damage threshold are not clear, however, because deleterious contaminants may be removed also.

An experiment was initiated, therefore, to (a) quantify the roughness increase with ultrasonic cleaning time and (b) determine the effect on laser damage threshold. Glass substrates were obtained which were ground and polished with materials not considered absorbing at a wavelength of 1.06 μm. After being appropriately characterized and ultrasonically cleaned, the substrates were to be damage tested using pulsed 1.06 μm laser irradiation.

SAMPLES

Three types of optical glass materials were prepared: Optosil I, BK-7, and commerical quartz. The test samples were initially measured for TIS surface roughness by NWC. Within each class of material and overall, the samples were all relatively smooth and exceptionally uniform in scatter. The RMS roughness values were as follows: 16.0 $\pm$ 0.2 Å (Optosil), 16.2 $\pm$ 0.4 Å (BK-7), and 16.4 $\pm$ 0.3 Å (commercial quartz). These measurements were made after the surfaces were flash-coated with silver to achieve high reflectivity. Afterwards, the silver was removed with a dilute nitric acid solution. This solution does not attack quartz and optical glasses.

PROCEDURES AND DESCRIPTIONS

Once the samples were characterized, the experimental procedure used was the following:

1. Prepare standard dilutions of three bath solutions. The solutions chosen were Micro, Radia, and distilled water.

2. Ultrasonically clean samples of the three glass materials according to Table 5.

TABLE 5. CLEANING SOLUTIONS VERSUS DURATIONS

Solution	5 min.	30 min.	120 min.
Micro	X	X	X
Radiac	X	X	X
Distilled H_2O	--	--	X

3. Make sure the bath covers the samples completely. Monitor both pH and temperature throughout the process. The chemical activity of Micro and Radiac depends on temperature, and the resultant changes in the glasses may depend on the acidity or alkalinity of the bath.

4. After each sample is removed from the bath, rinse it first in distilled water, then in alcohol, then rapidly blow the surface dry.

5. Send samples to the Naval Weapons Center for postcleaning characterization.

6. Damage test the ultrasonically treated samples and untreated control samples.

Micro and Radiac are commercially available, standar solutions for routine cleaning of laboratory glassware. Both materials are considered harmless to glasses, and both are considered to promote ultrasonic cleaning cavitation action -- chiefly because they increase wetting and decrease liquid surface tension. Standard dilution is three volumes of water to one volume of solution. For this experiment deionized, distilled water was used.

Micro is a mixture of complexing and sequestering agents, is a solubilizer, and contains both anionic and nonionic surface active agents. It acts in all of the chemical and physical ways involved in cleaning: wetting, solubilization, complexing, emulsification, dispersion, decomposition, antiredeposition, lowering of surface tension, softening, etc.

Radiac wash will sequester metallic ions. It lifts up and firmly suspends contaminating particles, allowing them to be rinsed away. It is a liquid compound (as is Micro); and it combines a number of chemical and physical principles which cause it to act as a surface-melting agent, chelater, carrier, ionexchanger, emulsifier, soluent, complexer, peptizer, and detergent.

Two kinds of monitor samples were made part of the total experiment. One type of monitor was the group of samples subjected to only the 2 hour distilled water cleaning treatment. Differences in final roughness values were expected to be maximum for the longest treatment period. Also, any significant differences could be expected to result from relative acidity or alleavity (i.e., the pH values). The other type of monitor was the group of samples chosen to be classical controls. That is, they were not ultrasonically cleaned at all. Each monitor group contained at least one sample of each type of optical glass material used in this experiment.

Ultrasonic cleaning was done in a Bransonic Model 52 device. This was the same cleaner, in fact, that was used to do the work reported in Reference 2.

RESULTS

Table 6 summarizes the ultrasonic cleaning conditions for the three baths used. They are included in this report mainly for completeness. Unfortunately, none of the ultrasonically cleaned samples exhibited new roughness values which differed significantly from their precleaning values. As a result, there was no point in doing any damage testing.

Changes in the samples did occur as a result of the cleaning treatments. Although the roughness values did not change to statistically different values, the standard deviations of the measurements did increase by about a factor of 10.

Earlier work (Ref. 2) indicated that factors of 4 increase in glass surface roughness were obtainable for 17.5 hours of ultrasonic cleaning. For 3/4 hours, the roughness changes were not statistically significant. In addition, the work of others (cited in Reference 2) had indicated significant changes in metal surface roughness for cleaning periods of 1 to 2 hours. It was felt likely that 3 hours could be sufficient to produce significant changes in some or all of the three glasses chosen for this experiment.

The chief difference in sample preparation between the present and earlier work was that the current samples had one polished face and one fine-ground face, whereas the earlier glass samples had two polished faces. Although the roughness values did not change, ultrasonic cavitation apparently did attack the three glasses--the scribed identification codes on the fine-ground faces were sometimes nearly obliterated regardless of which bath was used.

Perhaps polished glass surfaces are not readily attacked by ultrasonic cleaning, regardless of the type of bath used, as long as the duration of the cleaning operation is kept reasonably short. Certainly, times much less than several hours appear to be short enough.

TABLE 6. ULTRASONIC BATH PARAMETERS VERSUS TIME

Time (min)	Micro		Radiac		Water	
	Temp. (°C)	pH	Temp. (°C)	pH	Temp. (°C)	pH
0	19.0	9.9	16.6	5.1	19.1	7.7
5	19.5	9.9	18.1	5.1	--	--
30	23.8	9.9	24.2	5.1	25.4	7.8
60	28.4	9.9	98.5	5.1	28.7	7.9
90	30.2	9.9	30.6	5.1	30.3	8.0
120	31.5	9.9	32.0	5.1	30.9	8.1

IV CONCLUSIONS

ROUGHNESS INVESTIGATIONS

The surface statistics of many of the samples appeared to be dominated by the presence of particulate matter. These particles apparently accumulated over a period of time because earlier roughness values were generally smaller than later ones. Some samples could be cleaned of particles using only ionized dry nitrogen gas flowing across the surfaces. Other samples could only be cleaned using a mild soap and water wash. And some samples could not be stripped of particles at all. It seems clear that further experiments need to be done if one wishes to ascertain the origins and natures of the various types of particles implied by the observations of this project.

Although there may have been several types of particles, or even very deep holes, at the sample surfaces, relationship (1) does not seem to be sensitive (at least in exponent m) to those perturbations. The essential differences among the techniques used to infer roughness values did not appear to invalidate relationship (1) either. The finest probe was the Talystep SP method, in which the typical length is about 0.061μm. The next finest probe was the FECO method, in which the typical length is 1 to 2 μm. The SP and FECO methods can determine the actual surface height distribution as well as an RMS roughness value. Their data can be routinely analyzed to separate the abnormal heights (i.e., the particles) from the background heights; but the SP method can more easily yield slope distribution functions and autocovariance functions than can the FECO method. Finally, the TIS method is relatively crude (the typical length being 1 to 2 mm), is sensitive to the presence of scatter sites such as holes or particles (but cannot separate out their effects from the background), and theoretically requires the surface height distribution to be Gaussian and the RMS roughness to be very much smaller than the wavelength of the light used to make the measurements of scatter.

These considerations imply, then, that relationship (1) remains valid as an estimating tool for damage tests of arbitrarily-prepared window samples.

While surface roughness is an inferred quantity and not a well-defined property of the surface, it remains a very useful characterizaton parameter to relate with damage threshold.

ULTRASONIC CLEANING INVESTIGATION

The observations made concerning the long duration ultrasonic cleaning of polished glasses seem to be mostly encouraging. Even though fine-ground surfaces may suffer from cavitation attack, true windows will have two polished surfaces. Therefore, one may expect minimal changes to the optical surfaces of ultrasonically cleaned windows if the cleaning duration is very short.

There exists two other possibilites which might be important, but are not detectable in the design of the experiment reported here. One is that ultrasonic cleaning can easily attack ground surfaces, even for short cleaning times, and generate extensive (i.e., long-range) and detrimental structural changes in the window material. This has ramifications for windows with unpolished edges. The second possibility is that polished surfaces are attacked but in such a way that material is rather uniformly removed. The net effect would be minimal roughness and figure changes, but yet a component thickness change.

Dealing with the first possibility may require sophisticated techniques such as bending moment tests or optical stress measurement tests. The second possibility, considered to be very unlikely, is more easily handled. Half of the polished surface could be covered with a resilient material that would not suffer attack by the ultrasonic cleaning. One suggestion, made by Naval Weapons Center, is to use beeswax. Changes in the level of the exposed glass would be detectable optically (i.e., by interference fringe shift) or mechanically (i.e., by Talystep scanning).

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