

Laser-Gyro Materials Studies

by
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and
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Research Department

MARCH 1986

**NAVAL WEAPONS CENTER
CHINA LAKE, CA 93555-6001**



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FOREWORD

The report documents studies performed in support of the Advanced Technology Demonstration Laser-Gyro Program from 1984 to 1985. The work was carried out as part of a material characterization effort for laser-gyro materials funded by Naval Air Systems Command Project #137-831. The study consisted of measurements of material properties of low-expansion glass and glass-ceramic materials that have been candidates for use as laser-gyro bodies. The goal of this research effort was to evaluate the presently available glass-ceramic materials and to suggest which materials are best suited for laser-gyro applications.

This report was reviewed for technical accuracy by D. K. Burge.

The authors wish to thank Dr. J. E. Shelby, Alfred University, for the helium permeability measurements and Dr. S. F. Jacobs, Optical Sciences Center, University of Arizona, for the thermal expansion measurements.

Approved by
E. B. ROYCE, Head
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19 ABSTRACT (Continue on reverse if necessary and identify by block number) Material properties of low-expansion glass and glass-ceramic materials have been measured. The materials that have been characterized are ultralow-expansion (ULE) type 7971 quartz, a new glass-ceramic material RLA 559,122 from Corning Glass Works, fused quartz from General Electric, Zerodur from Schott Glaswerke, and Cervit C-101 from Owens-Illinois. Characterization has included measurements of X-ray powder diffraction patterns, some elemental analyses, helium permeability, thermal expansion, particle-size distributions, optical properties, and optical finish studies.					
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INTRODUCTION

Since production of a major domestic lithium-aluminum-silicate glass ceramic has ceased, there has been increased dependence on other lithium-aluminum-silicate glass ceramics such as Zerodur made by Schott Glaswerke, or ultralow-expansion (ULE) type 7971 quartz made by Corning Glass Works for applications requiring ultralow thermal expansion. Other requirements for ultraprecision measurement applications include low helium gas permeability and stability during thermal cycling in the -23 to 177°C temperature range. Neither Zerodur nor ULE quartz are acceptable materials for this range (References 1 through 3). Furthermore, the few available remaining lithium-aluminum-silicate glass-ceramic products are not of a reproducibly acceptable quality nor are they easily obtainable. Thus, there is a need for new oxide glass ceramics with ultralow expansion, low helium permeability, and dimensional stability over an extended temperature range.

A glass ceramic is a commercially available class of unique materials initially developed in the late 1950s at Corning Glass Works (Reference 4). They are polycrystalline solids prepared by the controlled crystallization of glasses. Crystallization is accomplished by subjecting suitable glasses to a carefully regulated heat treatment that results in the nucleation and growth of crystal phases within the glass. The degree of crystallinity can be varied from the amorphous glass at one extreme to the completely crystalline glass ceramic at the other extreme using identical chemical compositions. These glass ceramics are almost entirely free of porosity and are substantially harder and more abrasion resistant than most glasses (Reference 5).

Presently, one of the most important applications of glass ceramics is as containers for the laser discharge in ring laser gyroscopes used for inertial guidance systems. The primary requirement for an ideal laser gyro body is that it have a minimal thermal expansion coefficient. This will leave the resonant frequency of the optical cavity unchanged throughout the environmental temperature range encountered by an operational laser gyro.



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A summary of material properties of low-expansion glass and glass-ceramic materials will be presented. The materials that have been characterized and for which data are presented are ultralow-expansion (ULE) type 7971 quartz and a new glass-ceramic material RLA 559,122 from Corning Glass Works, fused quartz from General Electric, Zerodur from Schott Glaswerke, and Cervit C-101 from Owens-Illinois. Characterization has included measurements of X-ray powder diffraction patterns, some elemental analyses, helium permeability, thermal expansion, particle size distributions, optical properties, and optical finish studies.

EXPERIMENTAL MEASUREMENTS

Elemental analyses were performed on Cervit and Zerodur by Galbraith Laboratories, Inc., Knoxville, TN. Table 1 shows the compositions of these materials. ULE is a fused silicon oxide material doped with titanium oxide to form titanium silicate. No trace analysis is presented for the ULE material. We did not analyze the Corning glass-ceramic material because of an agreement with Corning Glass Works.

TABLE 1. Chemical Composition of Cervit and Zerodur.

Compounds	Cervit, %	Zerodur, %
Silicon dioxide (SiO_2)	72.47	55.50
Aluminum oxide (Al_2O_3)	18.06	25.30
Lithium oxide (Li_2O)	3.96	3.70
Titanium oxide (TiO_2)	1.81	2.30
Magnesium oxide (MgO)	0.93	1.00
Zirconium oxide (ZrO_2)	1.50	1.90
Zinc oxide (ZnO)	0.01	1.40
Phosphorous pentoxide (P_2O_5)	0.01	7.90
Miscellaneous oxides	1.01	0.95
Trace elements	0.11	0.01
Total	99.87	99.96

X-RAY ANALYSIS

The materials were analyzed by X-ray diffraction. Diffractometer scans were made on powders using nickel-filtered copper $\text{CuK}\alpha$ radiation. The instrument was a Philips diffractometer with a θ -compensating slit, diffracted beam monochromator, scintillator with pulse-height discrimination, and a copper source ($\text{CuK}\alpha = 1.5418$ angstroms, $\text{K}\alpha_1 = 1.5406$ angstroms; $\text{K}\alpha_2 = 1.5444$ angstroms).

The crystallite size was determined from slow-scan X-ray diffraction data taken on the (100) line. Measurements were made of the line width at half-maximum and used the Scherrer equation

$$D = \frac{k\lambda}{B \cos \theta}$$

where D is the crystallite size in angstroms, λ is the wavelength of the X-ray radiation, B is the width at half maximum in radians, θ is the diffraction angle, and k is the shape factor, chosen as 0.8 for these calculations.

A detailed explanation of the crystallite size calculation can be found in Reference 6.

An approximate value for the degree of crystallinity was obtained by performing a slow scan over the (100) peaks for all samples under the same conditions. The data were normalized by using an aluminum standard that was assumed to be 100% crystalline under the same conditions.

HELIUM PERMEABILITY AND DIFFUSIVITY MEASUREMENTS

The permeability and diffusivity of the glass ceramics were measured by the "membrane-permeation" method. Figure 1 shows a schematic diagram of the apparatus, which was developed at Sandia Laboratories, Livermore, Calif. First, the sample in the form of a thin disk is sealed between the O-rings of the upper and lower flanges. The spaces on both sides of the sample are evacuated, the sample is brought into equilibrium at the desired temperature, and helium gas is bled in on one side of the sample. The flow of gas through the sample is monitored as a function of time until a steady-state condition is attained. The gas is then pumped out, and the rate of gas evolution from the sample is recorded until it has decreased to a few percent of the steady-state flow rate. The determination of the permeability from the experimental data depends on the method used to monitor the gas flow through the sample. We used a mass spectrometer because other gases could leak into the system when using an ion gauge. The permeability was determined by direct measurement of the flow rate (as opposed to a pressure-rise rate) in which the permeating gas was constantly removed from the high-vacuum system by continuous pumping. The permeability K is derived from the relation $K = Bd/AP$, where K is the permeability expressed in atoms/sec-cm-atm, B is the flow rate, P is the pressure differential applied across the specimen, d is the sample thickness, and A is the area. Since a mass spectrometer does not yield an absolute flow-rate value, the sensitivity of the detection system must be calibrated by means of a flow-rate standard.

The diffusivity D is determined from the rate at which dissolved gas is evolved from the thin disk sample. After reaching a steady-state condition, the gas is rapidly pumped out of the gas-supply system. D , expressed in cm^2/sec , can then be found from the relation

$$D = \frac{L^2 \ln(B_1/B_2)}{\pi^2(t_2 - t_1)}$$

where B_1 and B_2 are the apparent flow rates at times t_1 and t_2 , respectively. This technique also yields a value for the solubility, which is equal to six times the total amount of gas evolved into the mass spectrometer after the diffusing gas has been removed from the high-pressure surface. Although there are several other techniques that have been used to determine gas diffusivity in glass, most studies have employed the method described above. In general, flow techniques have proven to be much more useful than sorption/desorption techniques because they offer simpler data analysis, better sample characterization, and greater accuracy. Consequently, they are more appropriate for studies of gas diffusion and solubility in glasses.

THERMAL EXPANSION

Thermal expansion was measured by an interferometric method in which the sample became the spacer in a Fabry-Perot interferometer, as described in detail in References 7 through 9. The light source was a frequency-stabilized, helium-neon laser; a sensitive photomultiplier detected changes in the cavity resonance frequency caused by changes in sample length L with temperature T . The cavity length (i.e., sample length) and cavity resonance frequency f are related by

$$\frac{\Delta f}{f} = \frac{\Delta L}{L}$$

At resonance, the cavity length is equal to an integral number m of half-wavelengths:

$$L = \frac{m\lambda}{2} = \frac{mc}{2f}$$

where c is the speed of light in vacuum. The thermal expansion coefficient α is

$$\alpha = \frac{1}{L} \frac{\Delta L}{\Delta T}$$

The ultimate precision in measuring $\Delta L/L$ depends on the frequency stability of the laser during the measurement. In this case, the uncertainty in α was $\pm 1 \times 10^{-10}/K$.

The optical properties of most of the materials are well known. The refractive index of all the materials is similar to that of fused quartz. Selected values for fused quartz are given in Table 2 (Reference 10). With the exception of the Corning experimental glass-ceramic material, opticians have used conventional polishing methods. To obtain supersmooth surfaces, variations of the bowl-feed polishing technique (Reference 11) with a pitch lap and abrasive in a water slurry have been used. In some optical shops, the polishing is done with a recirculating slurry. Although submerged polishing is easier to control than fresh-feed polishing (because the polishing rate is slower), the same low-scatter supersmooth surface finish also can be obtained in some cases by using fresh-feed polishing (Reference 12).

Recently, roughnesses as small as 2 angstroms root-mean-square (rms) have been measured on fused quartz and Zerodur surfaces polished by manufacturers of ring-laser gyros. The measurements were made at the Naval Weapons Center (NWC) using a Talystep surface-profiling instrument (Reference 13).

The main purpose of the optical study was to see if a new noncontact float-polishing process (Reference 14) would be suitable for producing supersmooth surfaces on fused quartz and glass-ceramic materials. For this study, groups of seven 1-inch-diameter samples of each material were blocked onto 4-inch-diameter glass plates using cyanoacrylate glue. They were prepolished to an optical flatness of about 1 to 2 fringes over the entire block and a surface roughness of about 12 to 15 angstroms rms as measured on the Talystep instrument. They were then taken to Osaka University, Japan, and polished under the direction of Prof. Y. Namba using a float-polishing machine designed by Prof. Namba and built by the Toyoda Company of Japan. The samples were inspected in a differential contrast microscope during the polishing experiments to determine at what point the scratches and other visible defects had been removed.

TABLE 2. Refractive Index
of Fused Quartz at 20°C
(Reference 10).

λ , μm	Refractive index
0.365015	1.474539
0.404656	1.469618
0.435835	1.466693
0.467816	1.464292
0.486133	1.463126
0.508582	1.461863
0.546074	1.460078
0.576959	1.458846
0.579065	1.458769
0.587561	1.458464
0.589262	1.458404
0.643847	1.456704
0.656272	1.456367
0.667815	1.456067
0.706519	1.455145
0.852111	1.452465
0.894350	1.451835
1.01398	1.450242

RESULTS AND DISCUSSION

X-ray powder diffraction data indicated that all glass ceramics could be indexed as partially crystallized and having the virgilite structure ($\text{Li}_x\text{Al}_x\text{Si}_{3-x}\text{O}_6$, where $x = 0.5$ to 1.0); this has been described as a stuffed, disordered β -quartz structure (Reference 15). The structure has a hexagonal unit cell with $a = 5.132(1)$ angstroms and $c = 5.454(1)$ angstroms (Reference 16). Table 3 summarizes the crystallite sizes of various commercially available β -quartz lithium-aluminum-silicate glass ceramics after ceramitization. Figures 2 and 3 show thermal expansion coefficients of some of the materials. Helium permeability data are shown in Figures 4 and 5.

TABLE 3. Crystallite Sizes and Percent Crystallinity of Laser Gyro Materials.

Material	Crystallite size, $\pm 100 \text{ \AA}$	Crystallinity, $\pm 10 \text{ vol\%}$
Zerodur	800	79
Quartz	500	53
Cervit C-101	1400	100
Corning RLA 559,122	650	70

In the optical studies, surface profile measurements were made before and after float polishing to show the improvement in the surface finish. Figures 6 and 7 show such profiles for fused quartz. This material was used as a reference because the float-polishing technique had been used previously on fused quartz, and it was known that a very smooth surface finish could be obtained.

Figures 8 and 9 show the improvement in the surface finish as a function of time for Zerodur and the new Corning glass-ceramic material, respectively. Figures 10 through 13 show surface profiles for Zerodur taken before and after float polishing. Similar profiles for the Corning glass ceramic are shown in Figures 14 through 17. Roughness measurements on the smoothest parts of these surfaces after float polishing gave values of about 2 angstroms rms, in good agreement with values measured on conventionally polished Zerodur and fused quartz samples. Thus, the new Corning glass-ceramic material has good potential for use in laser gyros.

Unfortunately, the 1-inch-diameter Cervit samples were damaged during the polishing studies, so they could not be measured. However, a 4-inch-diameter piece of Cervit was also float polished. Over the longest profiles (628 micrometers), the surface was slightly wavy, as shown in Figure 18. However, over shorter profile lengths, the surface roughness was less than 2 angstroms rms on the smoothest parts of the surface measured.

The 1-inch-diameter ULE quartz samples were also damaged during the float-polishing experiments. However, earlier experience at the NWC Optics Shop has indicated that these materials polish in a way similar to that of fused quartz. Thus, it is assumed that extremely smooth low-scatter surfaces also can be produced on this material.

CONCLUSIONS

Properties have been measured on ultralow-expansion (ULE) type 7971 quartz, a new glass-ceramic material RLA 559,122 from Corning Glass Works, fused quartz from General Electric, Zerodur from Schott Glaswerke, and Cervit C-101 from Owens-Illinois. Characterization has included measurements of X-ray powder diffraction patterns, helium permeability, thermal expansion, particle-size distributions, and optical properties, as well as elemental analyses and optical finish studies.

X-ray powder diffraction patterns showed all glass-ceramic samples to be partially crystallized and to have the virgilite structure. Particle-size data show that Cervit C-101 is 100% crystalline, Zerodur and the Corning material are about 75% crystalline, and fused quartz is only about 50% crystalline. Zerodur, Cervit, and the Corning glass ceramic were shown to have low helium permeability, while ULE and fused quartz have high permeability. All glass ceramics have thermal expansion coefficients in the low-expansion range, with α varying from $\pm 10^{-8}$ to 10^{-6} in the 0- to 600-K temperature range. Roughness measurements were performed on the new Corning glass ceramic, Zerodur, and Cervit after float polishing. The Corning material had a roughness of about 2 angstroms root-mean-square (rms), in good agreement with roughnesses measured on conventionally polished Zerodur and fused quartz samples. Cervit roughnesses were somewhat varied: Over the longest profiles (628 micrometers), the surface was slightly wavy; over shorter profile lengths, the surface was very smooth (~ 2 angstroms rms). ULE quartz samples also can be polished to a good, smooth, low-scatter surface finish.

Based on the data presented in this report, Cervit, Zerodur, and RLA 559,122 from Corning Glass Works have acceptable material properties for laser-gyro applications. It is hoped that further developmental research will enable all of these glass ceramics to be commercially produced.

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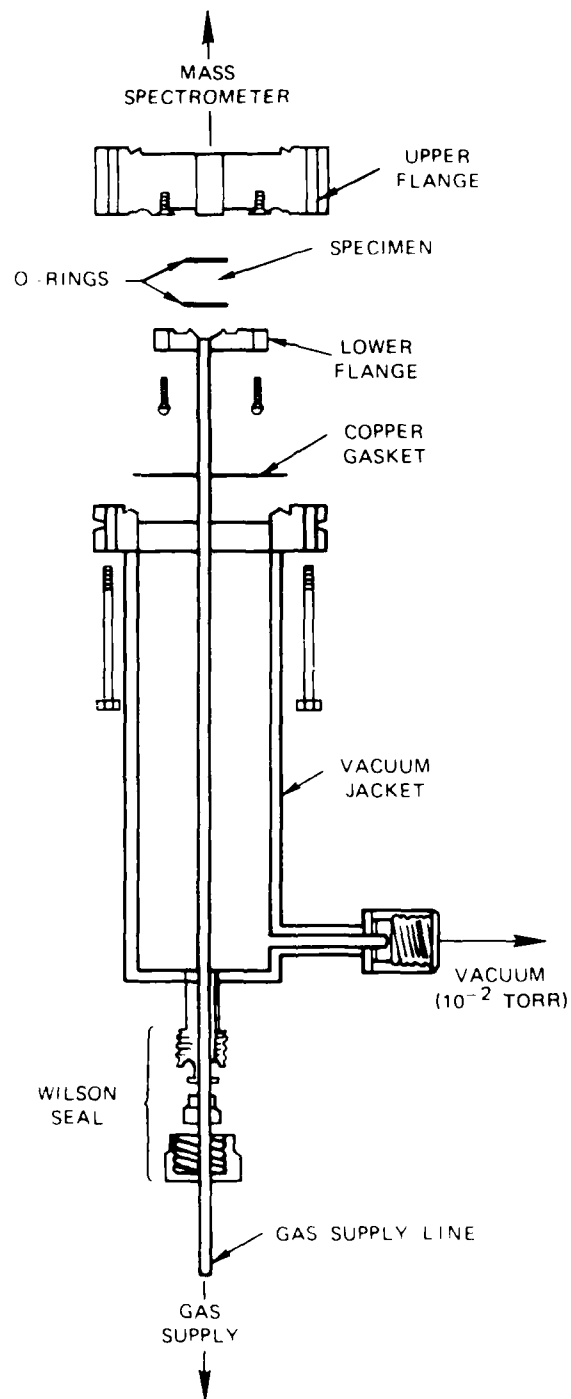


FIGURE 1. Schematic Diagram of Apparatus Used to Measure Helium Permeability and Diffusivity (After Shelby, Reference 2).

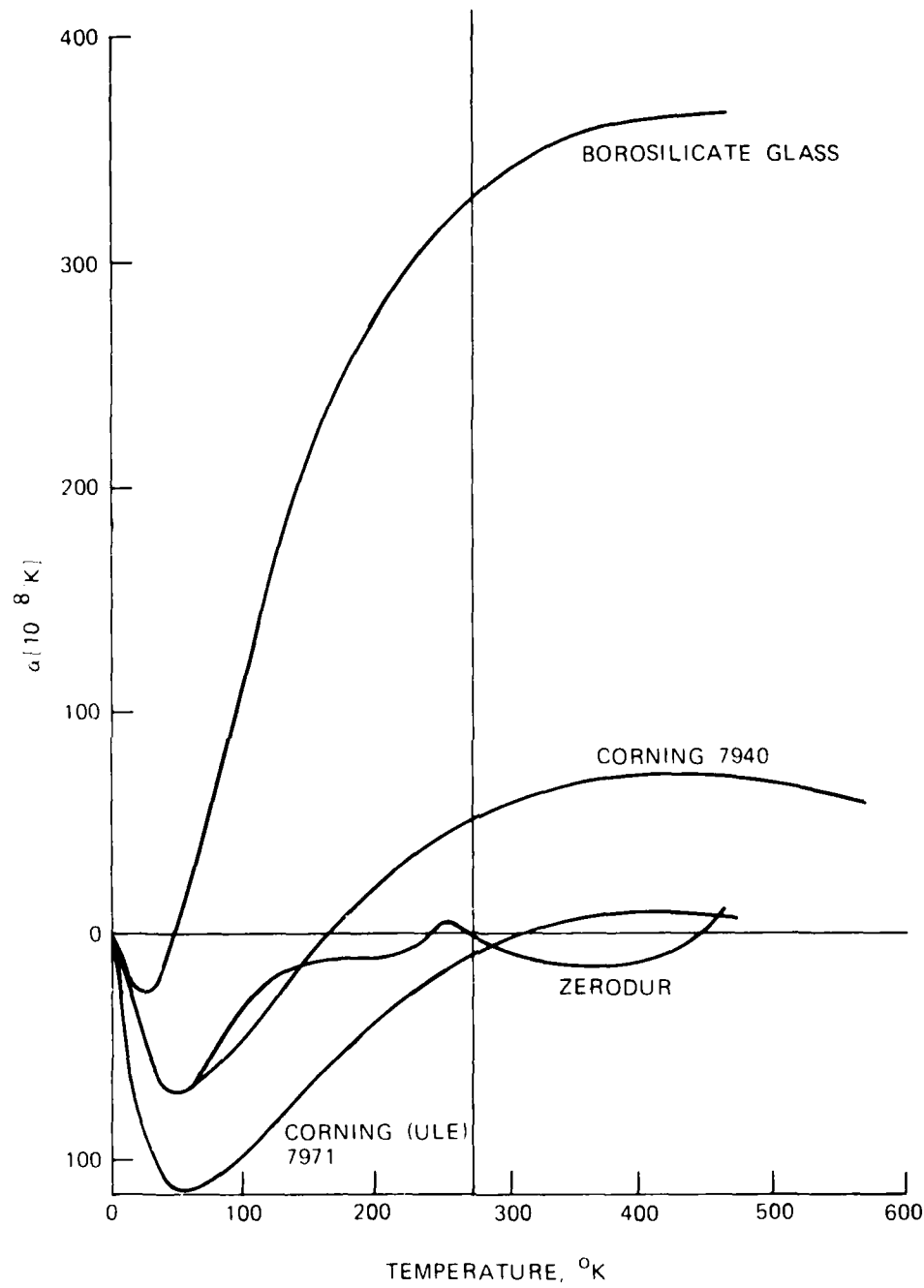


FIGURE 2. Measured Thermal Expansion Coefficient for Various Optical Materials.

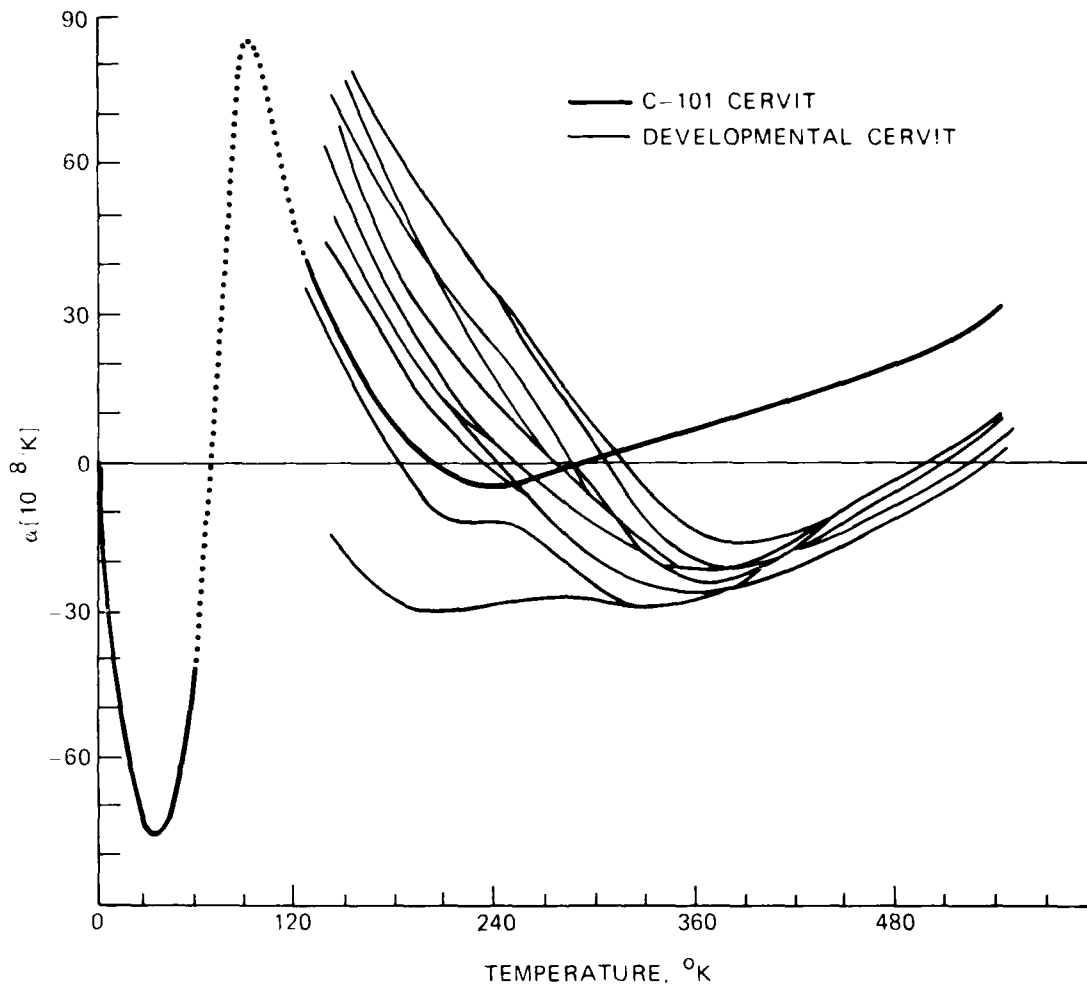


FIGURE 3. Measured Thermal Expansion Coefficient for Cervit C-101 and Developmental Cervit.

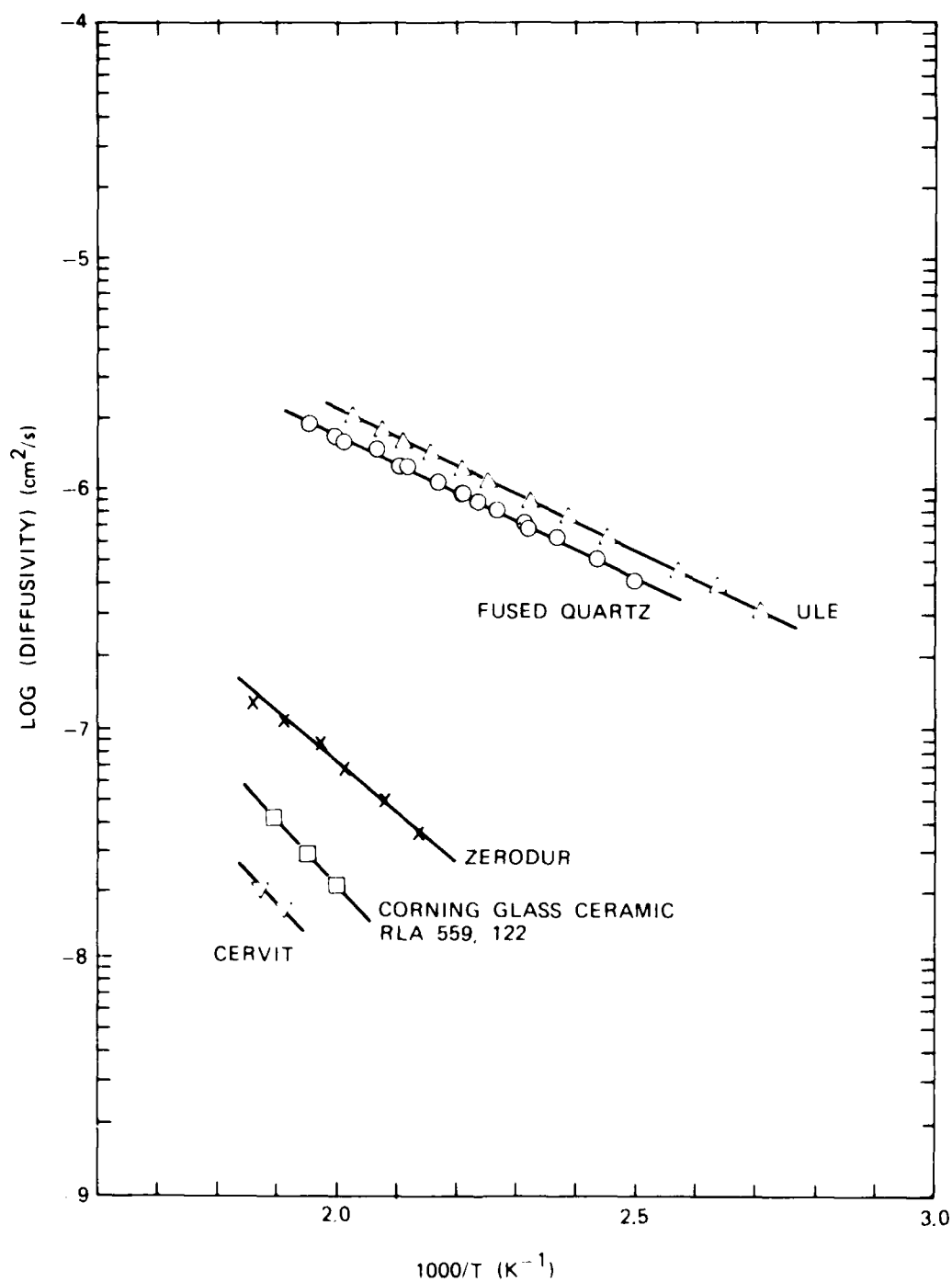


FIGURE 4. Measured Helium Diffusivity for Various Optical Materials.

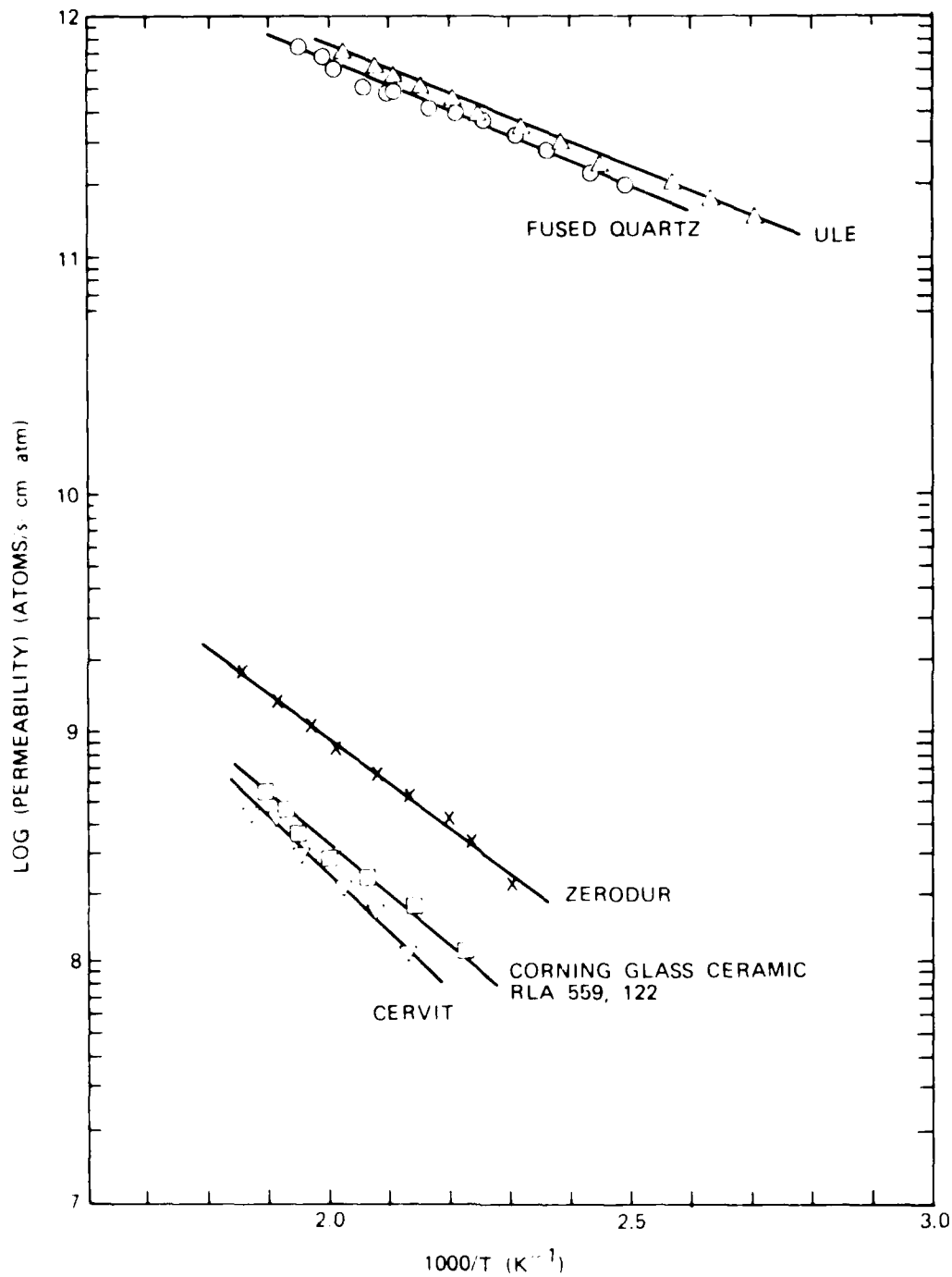


FIGURE 5. Measured Helium Permeability for Various Optical Materials.

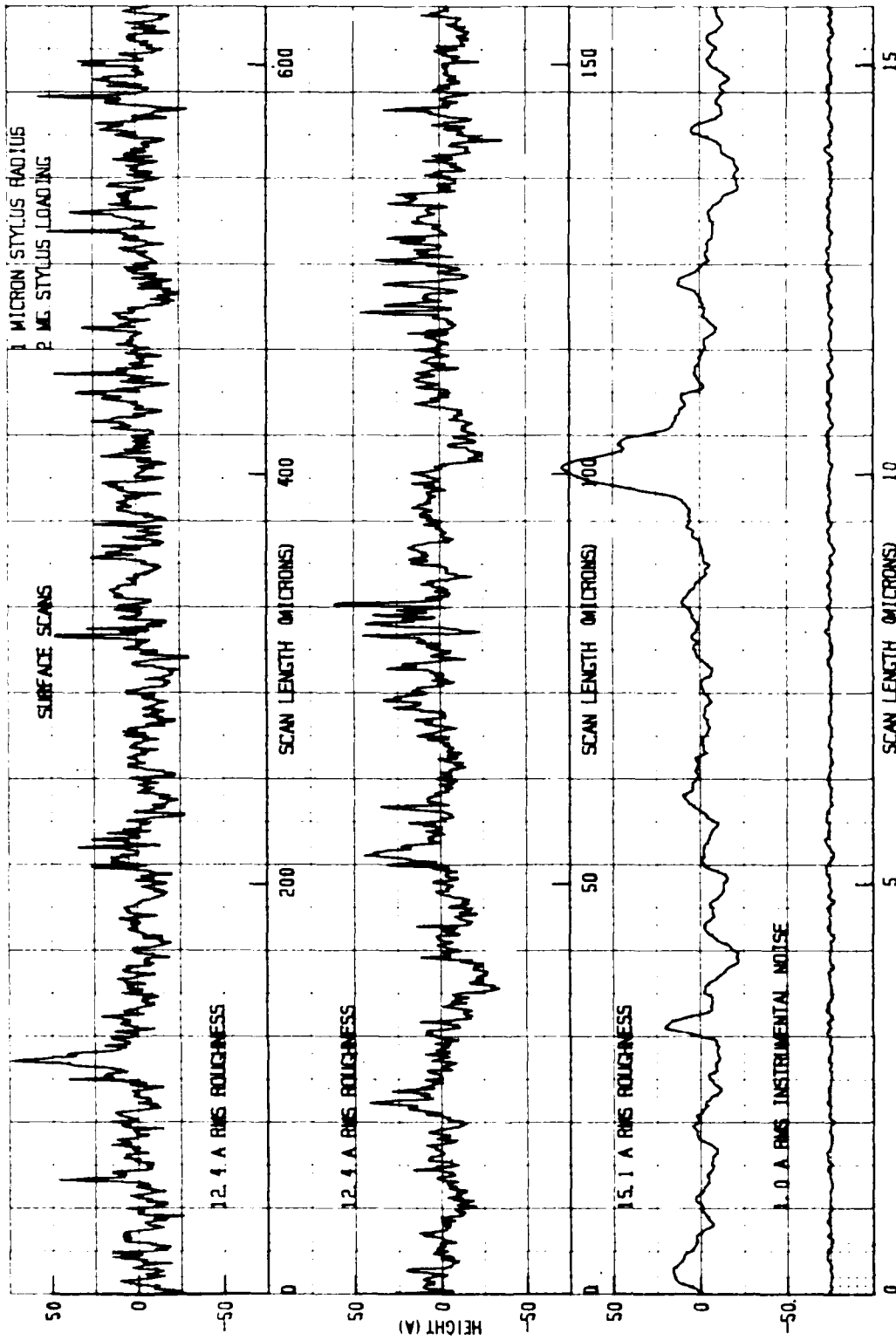


FIGURE 6. Measured Surface Profiles of Fused Quartz Before Float Polishing.

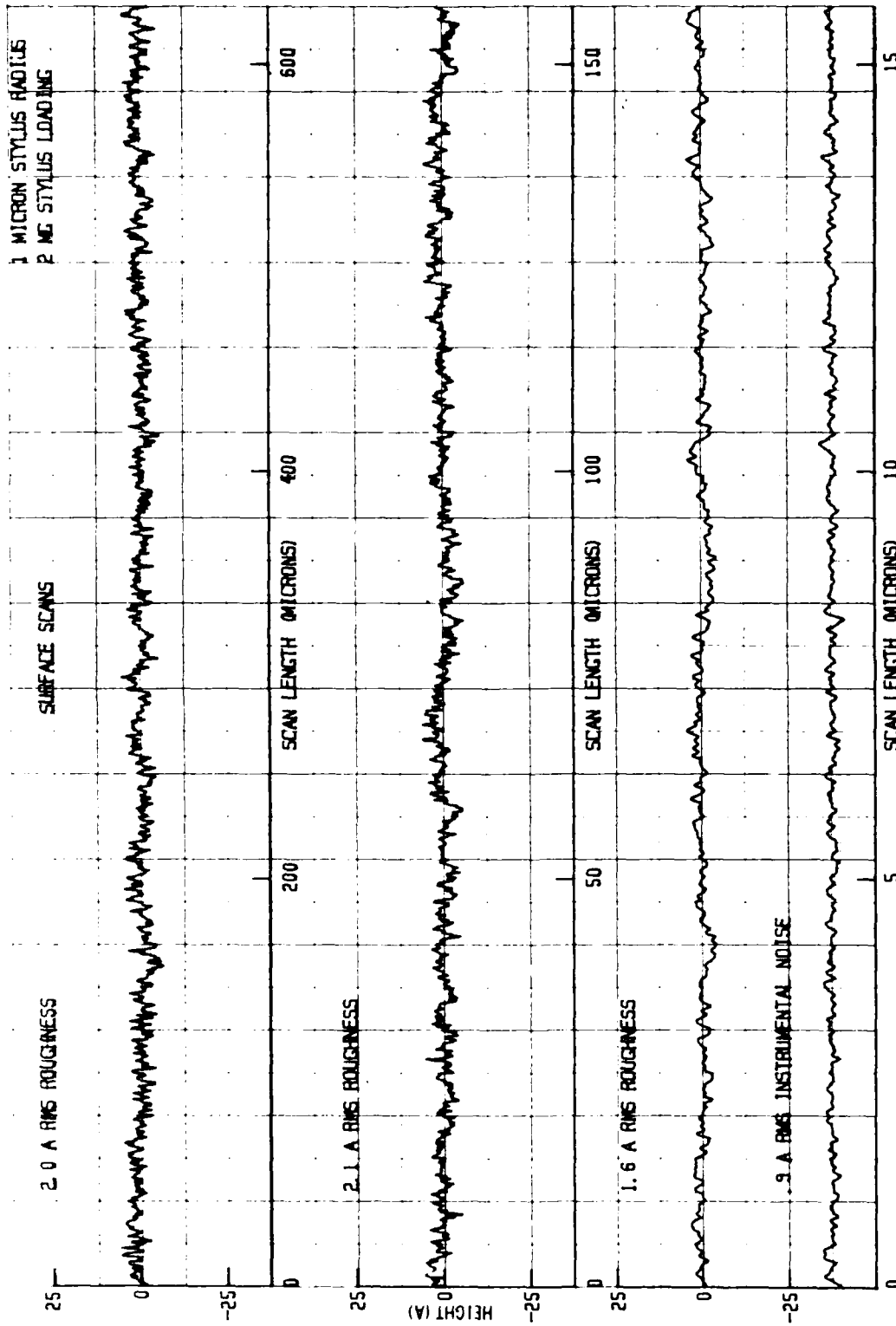


FIGURE 7. Measured Surface Profiles of Fused Quartz After Float Polishing.

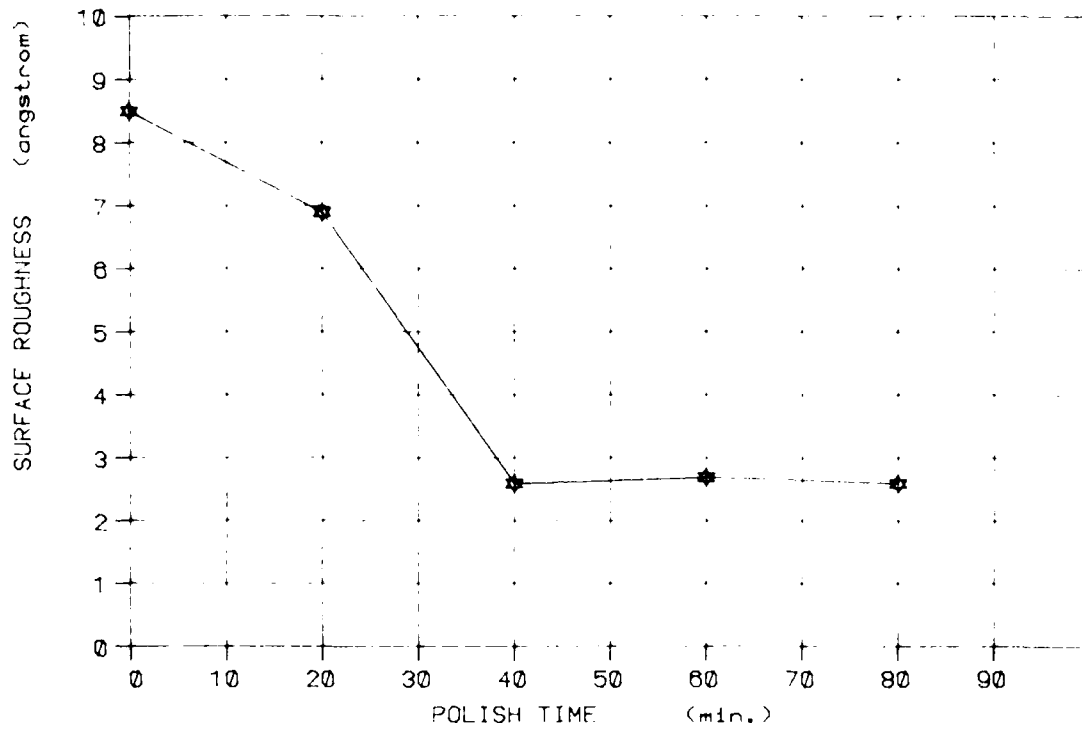


FIGURE 8. Measured Change in Surface Roughness as a Function of Float-Polishing Time for Zerodur.

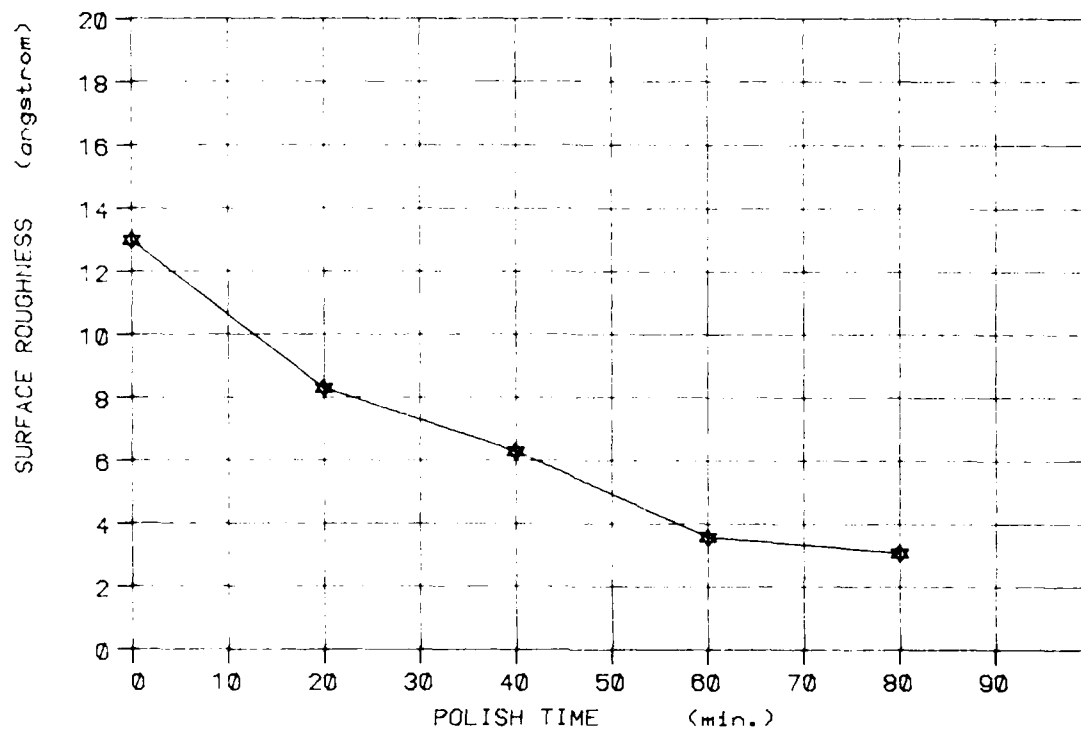


FIGURE 9. Measured Change in Surface Roughness as a Function of Float-Polishing Time for Corning Glass Ceramic.

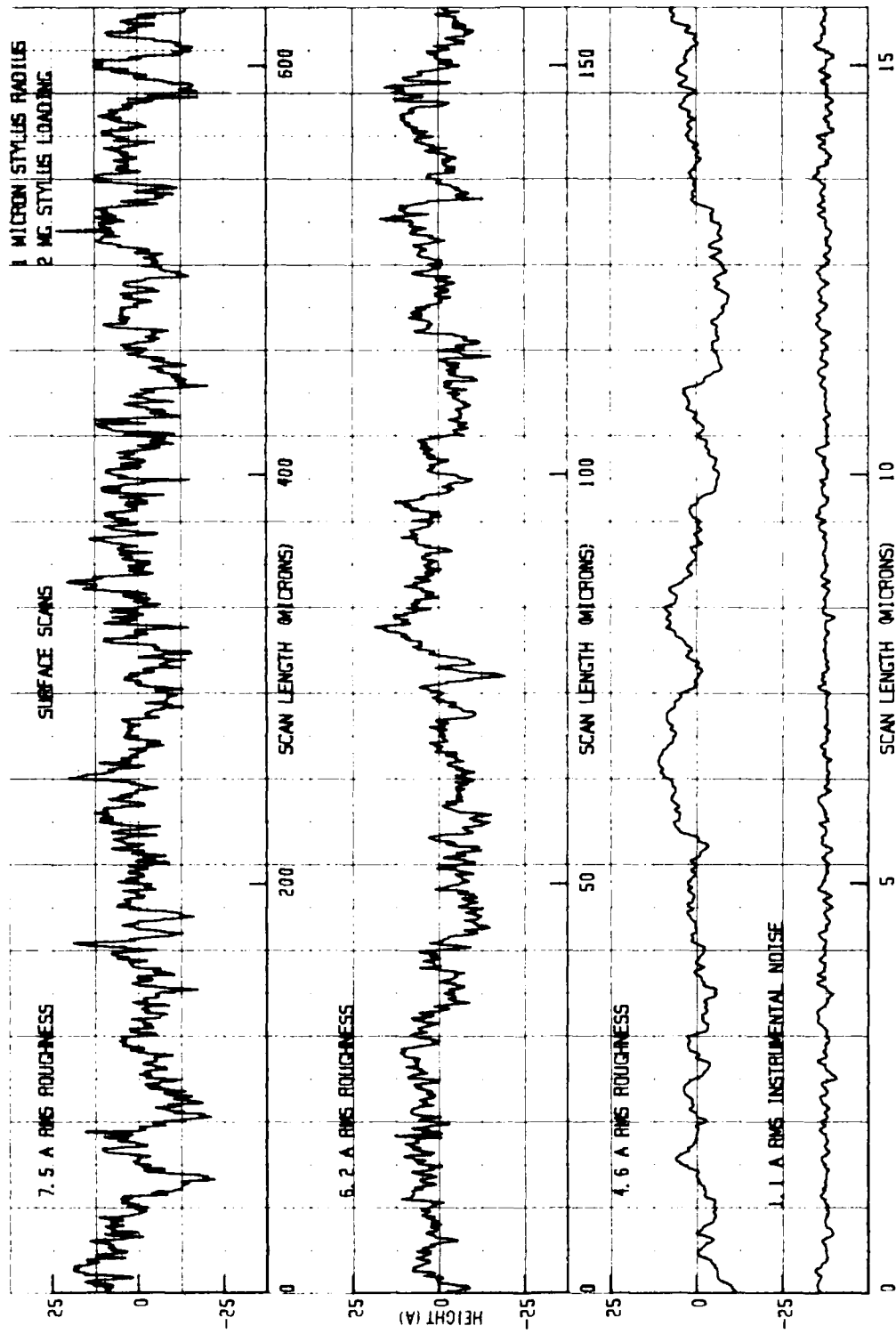


FIGURE 10. Measured Surface Profiles of Zerodur, Region 1, Before Float Polishing.

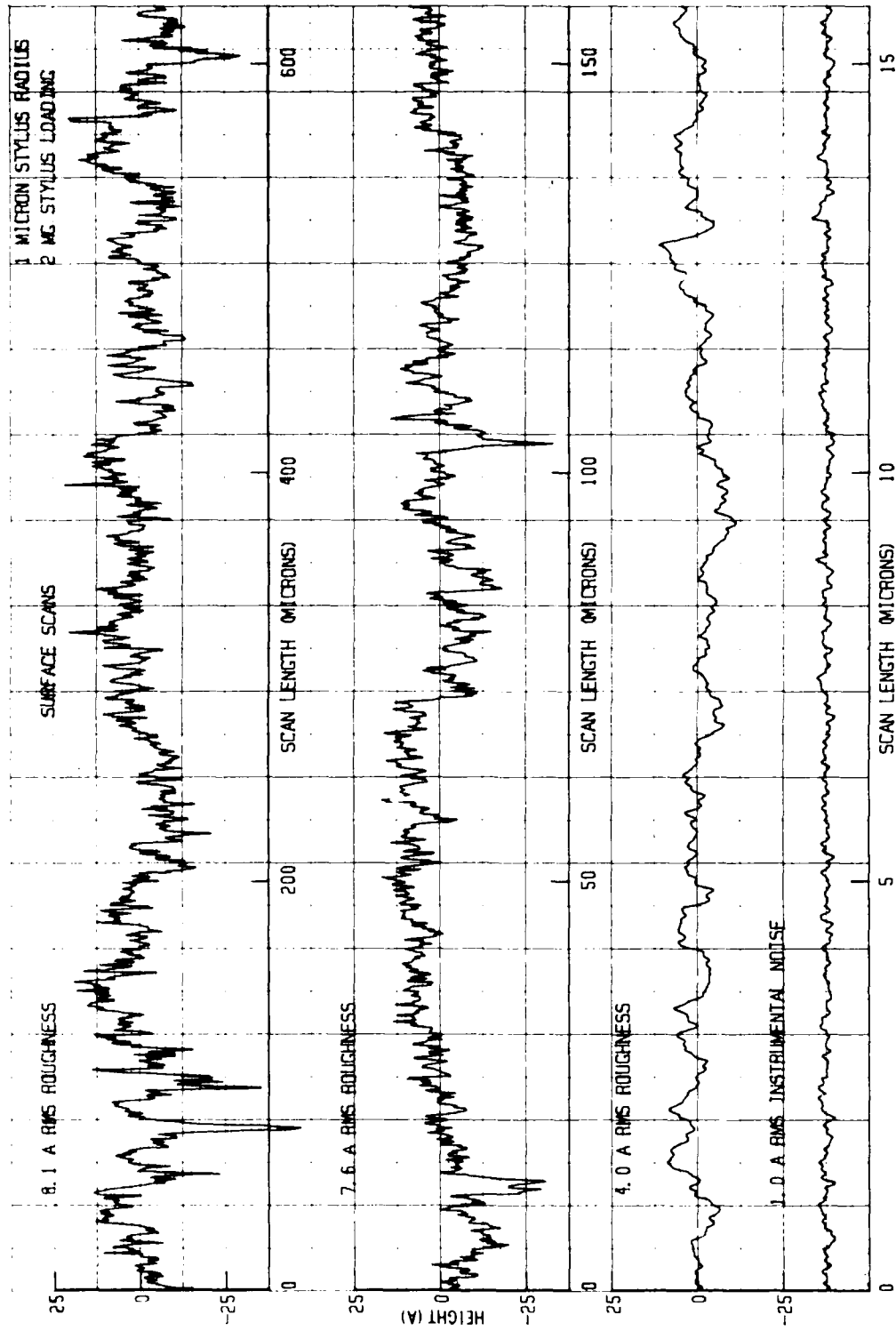


FIGURE 11. Measured Surface Profiles of Zerodur, Region 5, Before Float Polishing.

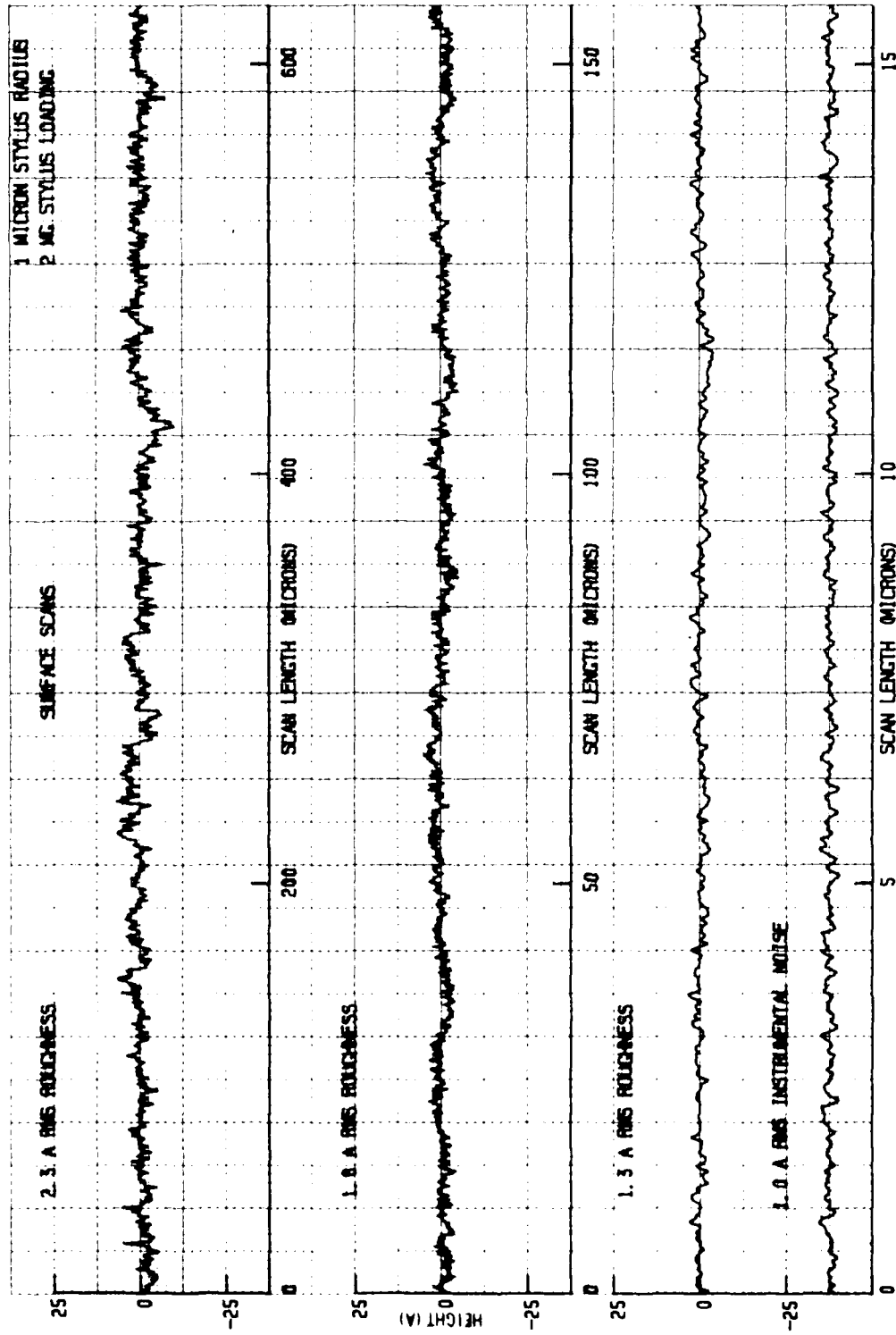


FIGURE 12. Measured Surface Profiles of Corning Glass Ceramic RLA 559,122, Region 1, After Float Polishing.

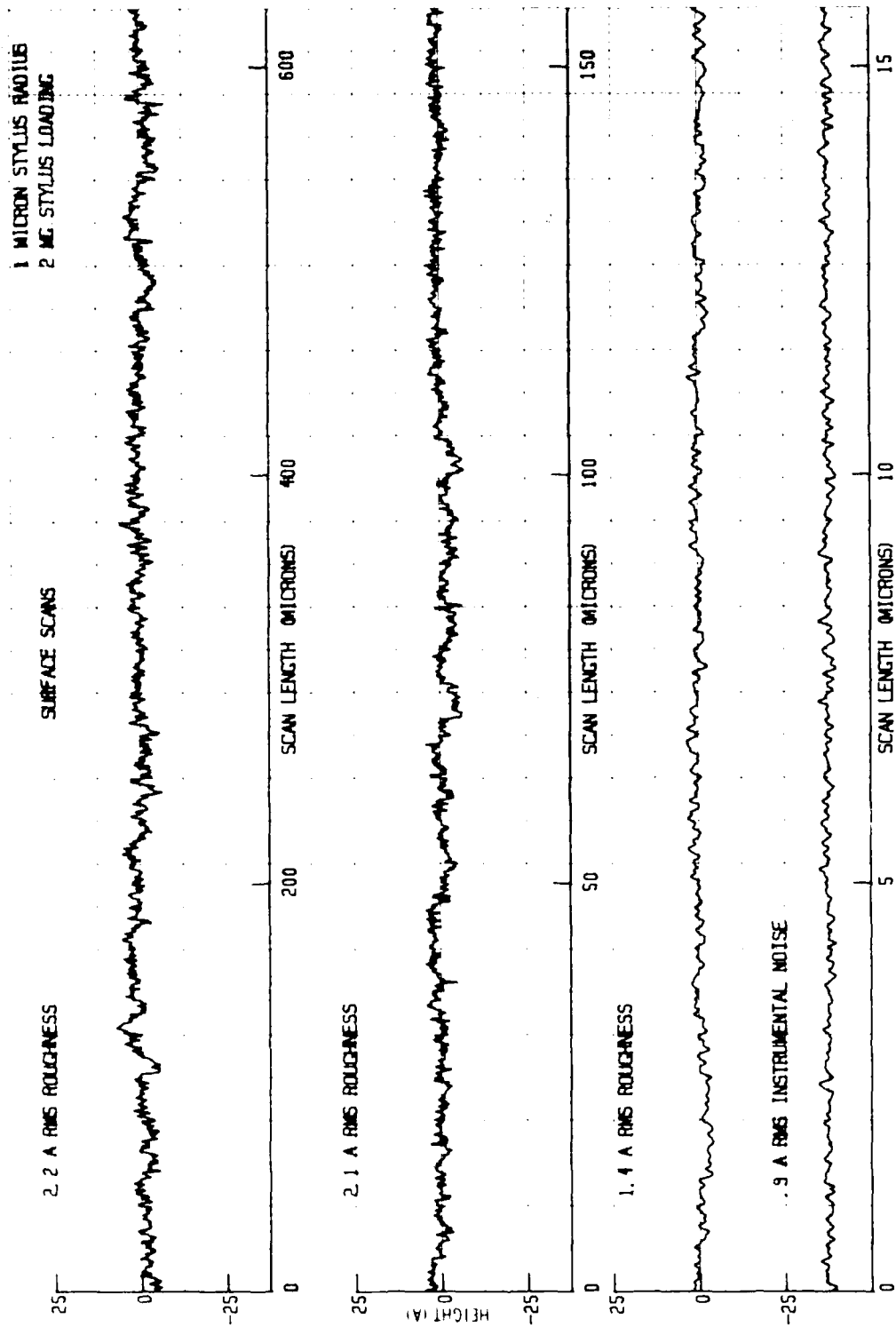


FIGURE 13. Measured Surface Profiles of Zerodur, Region 5, After Float Polishing.

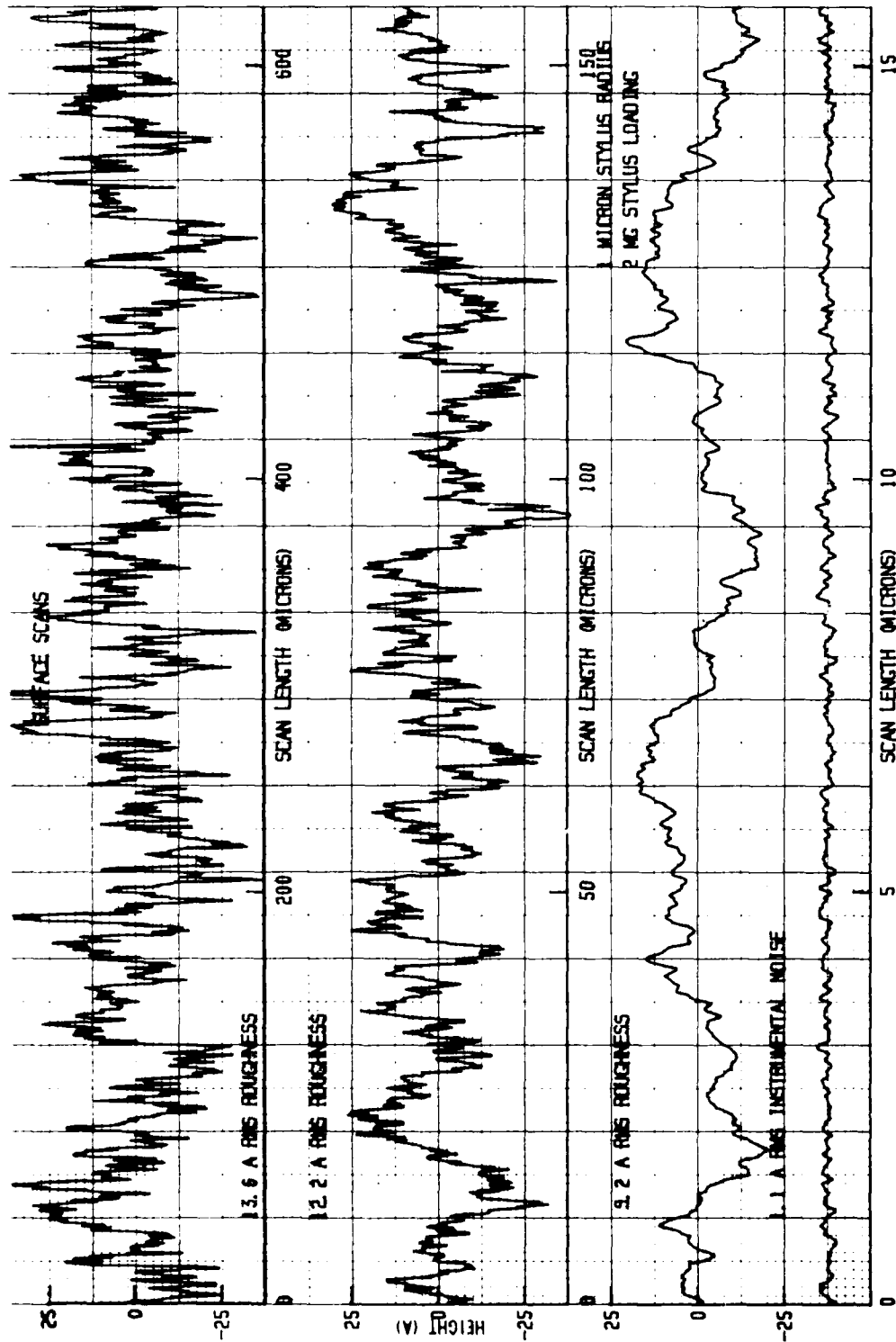


FIGURE 14. Measured Surface Profiles of Corning Glass Ceramic RLA 559,122, Region 1, Before Float Polishing.

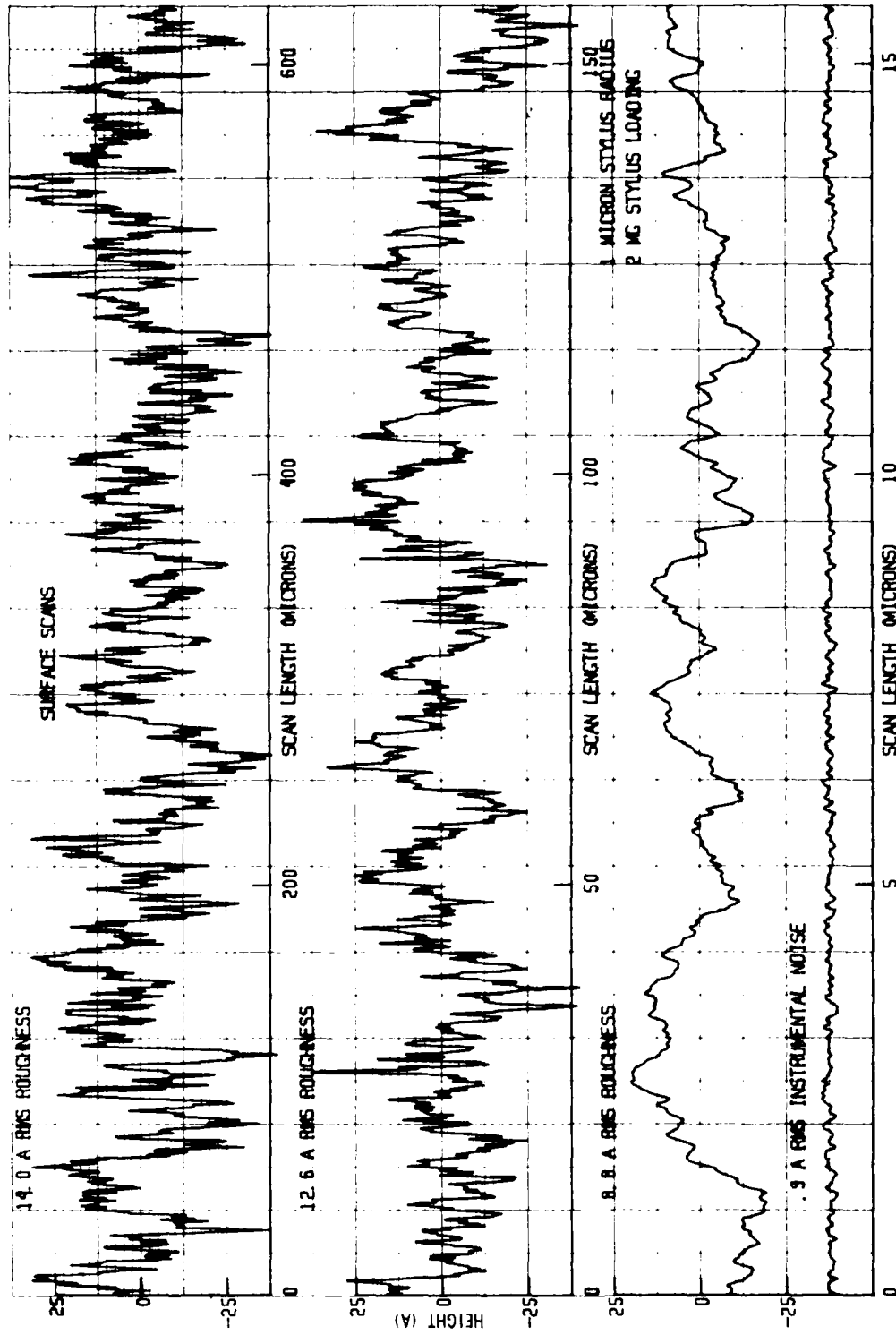


FIGURE 15. Measured Surface Profiles of Corning Glass Ceramic RLA 559,122, Region 5, Before Float Polishing.

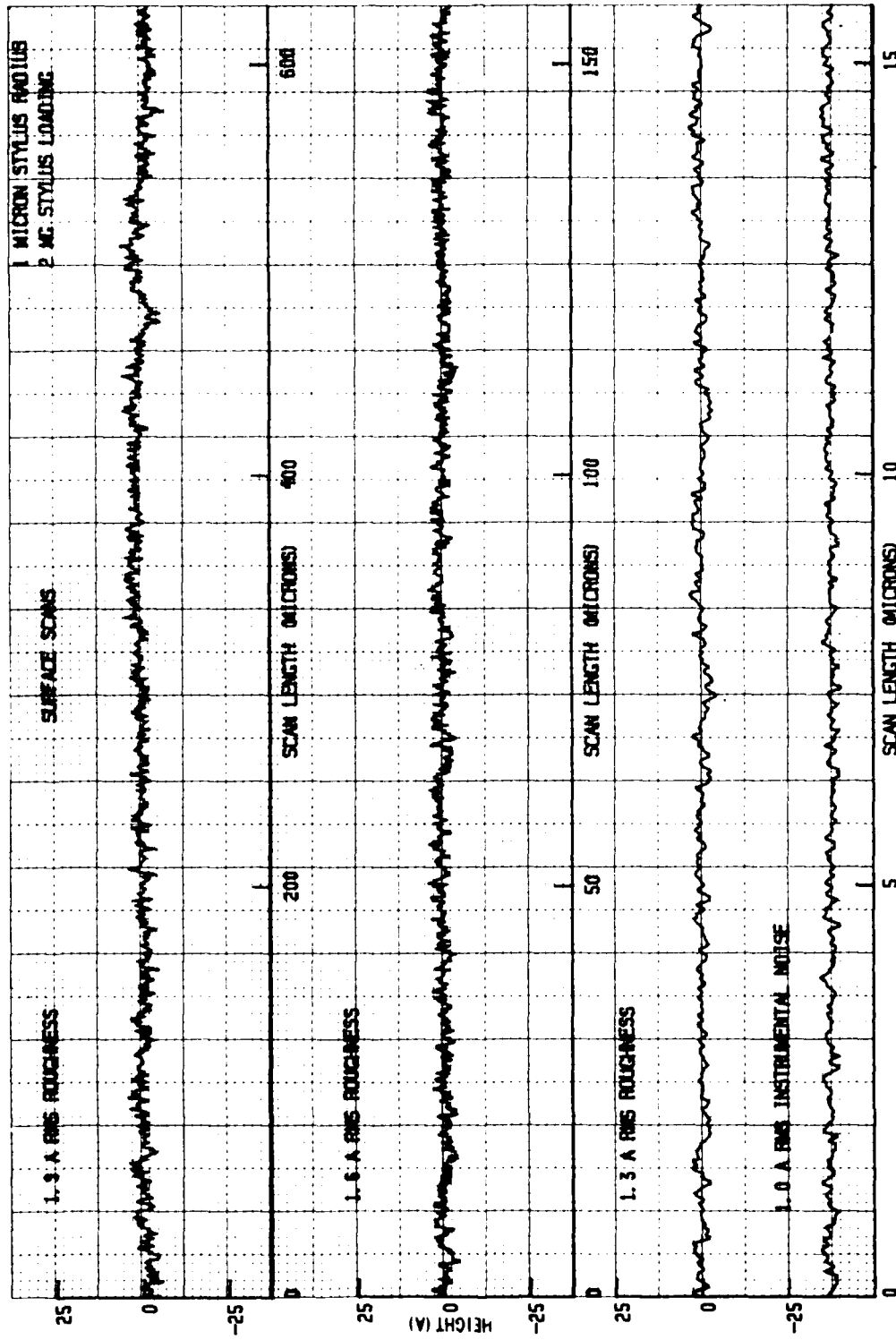


FIGURE 16. Measured Surface Profiles of Corning Glass Ceramic RLA 559,122, Region 1, After Float Polishing.

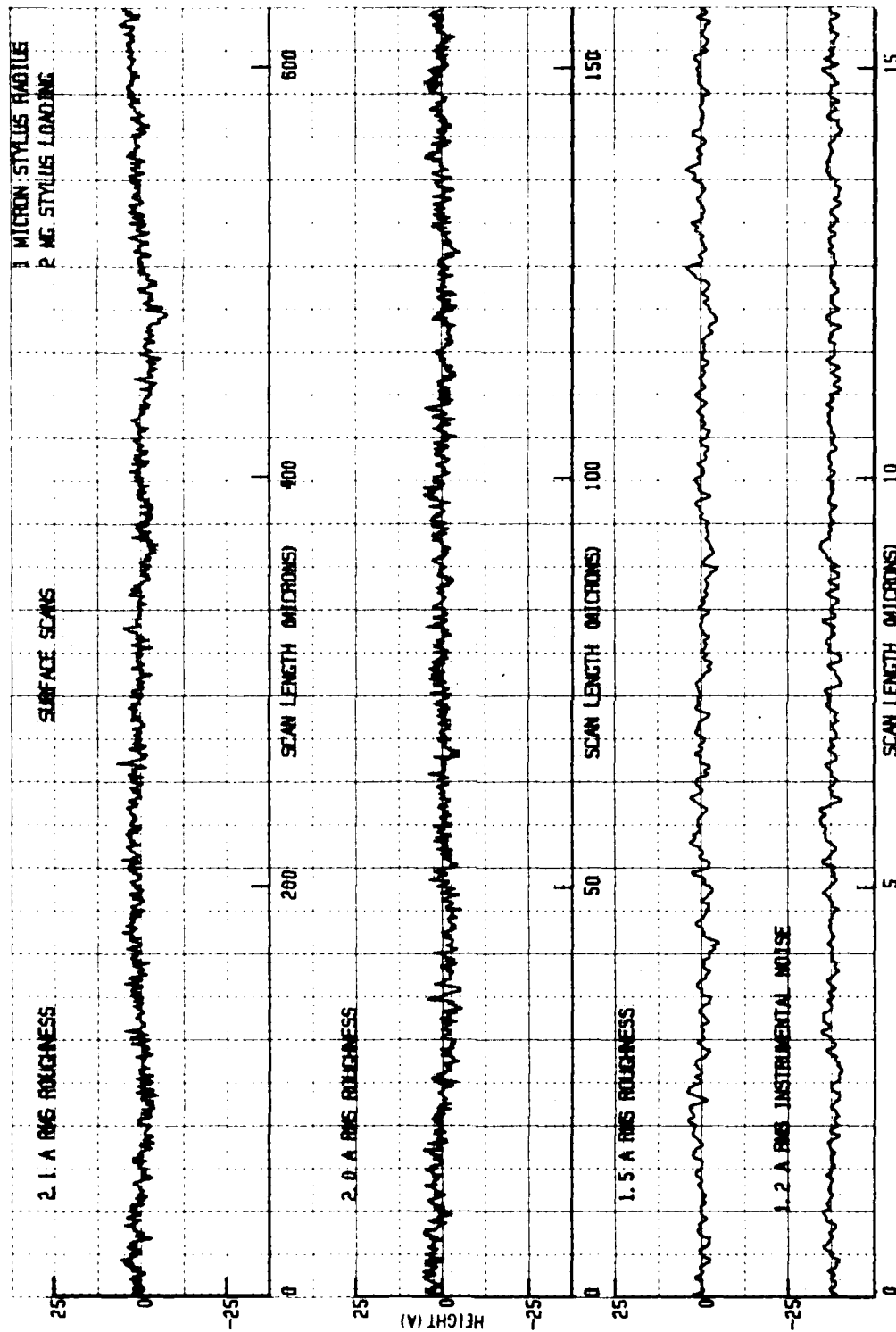


FIGURE 17. Measured Surface Profiles of Corning Glass Ceramic RLA 559,122, Region 5, After Float Polishing.

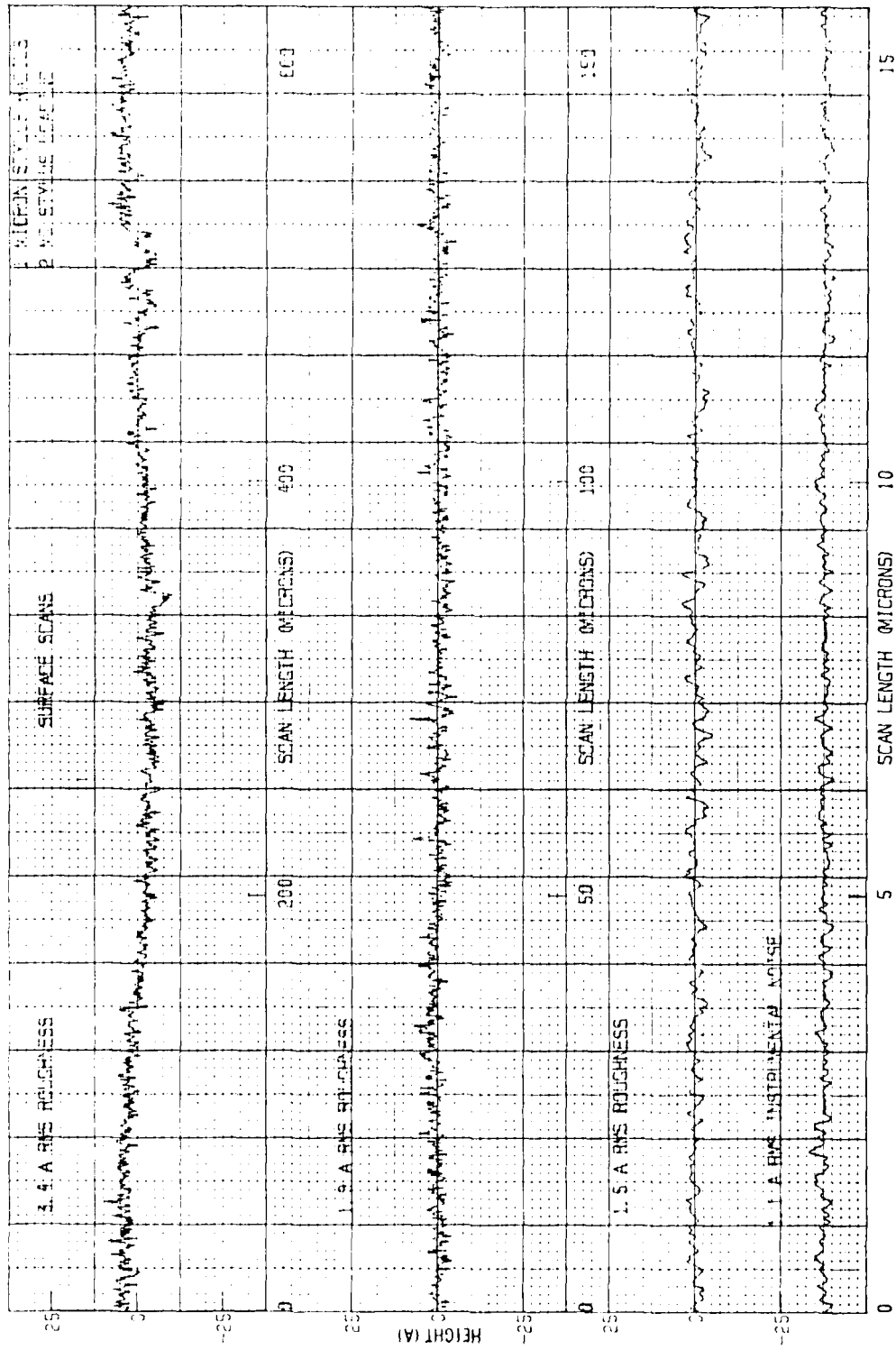


FIGURE 18. Measured Surface Profiles of 4-Inch-Diameter Cervit After Float Polishing.

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- 1 Air Force Armament Division, Eglin Air Force Base (AFATL/DLMA, R. McAdory)
- 1 Air Force Intelligence Service, Bolling Air Force Base (AFIS/INTAW, Maj. R. Lecklider)
- 12 Defense Technical Information Center
 - 1 Corning Glass Works, Corning, NY (M. Taylor)
 - 1 Honeywell, Inc., Avionics Division, Minneapolis, MN (T. Podgorski)
 - 1 Kimble Applied Research and Development, Toledo, OH (T. Broch)
- 6 Litton Systems, Inc., Woodland Hills, CA
 - F. DeLaat, M/S 3 (1)
 - J. Fischer, M/S 3 (1)
 - S. Hammons, M/S 19 (1)
 - T. Hutchings, M/S 12 (1)
 - J. Rahn, M/S 12 (1)
 - J. Todd, M/S 96 (1)
- 1 Rockwell International Corporation, Autonetics Division, Anaheim, CA (V. Sanders)
- 2 Singer Company, Kearfott Division, Little Falls, NJ
 - B. Leung (1)
 - M. Tarasevich (1)