NASA Technical Memorandum 101620

OPTICAL PROPERTIES OF SPUTTERED ALUMINUM ON GRAPHITE/EPOXY COMPOSITE MATERIAL

WILLIAM G. WITTE, JR.
LOUIS A. TEICHMAN

(NASA-TM-101620) OPTICAL PROPERTIES OF SPUTTERED ALUMINUM ON GRAPHITE/EPOXY COMPOSITE MATERIAL (NASA. Langley Research Center) 25 p CSCL 13I
N89-26245
Unclas
G3/37 0223905

AUGUST 1989

National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665-5225

Optical Properties of Sputtered Aluminum on Graphite/Epoxy Composite Material

William G. Witte, Jr. and Louis A. Teichman

ABSTRACT

Solar absorptance, emittance, and coating thickness were measured for a range of coating thicknesses from about 400Å to 2500Å. The coatings were sputtered from an aluminum target onto 1-inch-diameter substrates of T300/5209 graphite/epoxy composite material with two different surface textures. Solar absorptance and emittance values for the specimens with the smooth surface finish were lower than those for the specimens with the rough surface finish. The ratio of solar absorptance to emittance was higher for the smooth specimens, increasing from 2 to 4 over the coating thickness range, than for the rough ones, which had a constant ratio of about 1. The solar absorptance and emittance values were dependent on the thickness of the sputtered coating.

INTRODUCTION

Graphite reinforced resin matrix composites are currently being considered for use in large space structures because of such attractive features as light weight, high stiffness, and low thermal expansion. However, these materials must be protected against degradation by the various elements of the natural space environment, and must have the proper optical properties to minimize the thermal extremes to which the composite structure is subjected as the spacecraft moves in and out of the Earth's shadow. One way to accomplish this is to apply a protective coating to the bare composite which will resist degradative factors and have the proper ratio of solar absorptance (α_s) to thermal emittance (ϵ). The Materials Division of NASA Langley Research Center is investigating various methods of applying thermal control coatings to composite structures, such as anodizing and sputtering. This report documents some of the results obtained with magnetron sputtered aluminum on graphite/epoxy substrates.

Analytical studies (ref. 1) have shown that an uncoated graphite/epoxy surface has an α_s/ϵ of 0.85/0.85 and that a structure fabricated of this material will experience temperature extremes of -150 F to 175 F in low Earth orbit. Experimental studies (refs. 2-4) have shown that these composites will microcrack at these extremes. The protective coating must therefore modulate the temperature extremes to acceptable levels by judicious alteration of the solar absorptance and thermal emittance. An α_s between about 0.15 and 0.30 and an ϵ of about 0.65 and higher will produce an acceptable temperature range of about -100 F to 25 F for low Earth orbit (ref. 1).

References 5 through 8 describe techniques that have been used to control surface roughness and to texture surfaces for various objectives (e.g. better adhesion, appearance, optical storage of information by laser-melting small reflective spots on antireflective coatings, and solar energy thermal conversion). Many studies have shown that optical reflectance decreases with surface roughness (refs. 9-12). Techniques for texturing metallic and dielectric surfaces include chemical vapor deposition (CVD), chemical etching, and sputtering (refs. 13 and 14). In the case of composite laminates, surface roughness is controlled mainly by the texture of the caul plates, separation sheets, and bleeder cloths used during fabrication. References 15 and 16 present typical lay-up processing arrangements. The equipment that was used for fabrication in the present study produced a laminate that had a "rough" side with an average roughness of 170 microinches and a "smooth" side with an average roughness of about 25 microinches. Specimens of both surface finishes were sputter coated with aluminum for different lengths of time resulting in coating thicknesses ranging from 420Å to 2520Å.

TEST SPECIMENS

Specimen Description*

The specimens were 1-inch-diameter discs cut with a diamond impregnated core drill from a 50- × 26-inch laminate of 8 ply $[0,0,0,90]_s$ T300/5209 graphite/epoxy composite material. T300/5209 is a composite material composed of Union Carbide Thornel 300 PAN fiber impregnated with Narmco Materials 5209 epoxy resin. The laminate was fabricated at the Langley Research Center from unidirectional commercial prepreg. The laminate was layed-up on a smooth surfaced Teflon caul plate with a standard textured bleeder cloth on top, so that the cured composite had a smooth side and a rough side. Figure 1 shows SEM (Scanning Electron Microscope) photomicrographs of the two surfaces. Figure 2 shows traces of the surface roughness obtained with a Federal Products Corp. Model 2000 Surface Analyzer. Peak-to-trough measurements of the surface profile made along the striations of the smooth surface are in the range of 20 to 80 microinches. These striations are impressions caused by the surface finish of the Teflon caul plate. Roughness averages (Ra) were determined from traces obtained from the surface profiles using a waviness cutoff filter of 0.030 inch. This filter allowed traces of coarse irregularities with frequencies of less than 0.030 inch to be made but filtered out higher frequencies. The roughness averages obtained along and across the striations were 21 and

---

*Certain commercial materials and products are identified herein in order to specify adequately which materials and products were investigated in the research effort. In no case does such identification imply recommendation or endorsement of the product by NASA, nor does it imply that the materials and products are necessarily the only ones or the best ones available for the purposes. In many cases equivalent materials and products are available which could produce equivalent results.

29 microinches, respectively. The photo in figure 1 of the rough surface formed by the bleeder cloth shows the indentations in the resin formed by the warp and the woof of the cloth. The surface profile of the rough surface in figure 2 shows peak-to-trough measurements of up to 1600 microinches in variation. The average roughness was 170 microinches.

Specimen Preparation

After the specimens were cut from the sheet of T300/5209, they were lightly sanded around the edges with 600A WT silicon carbide paper to remove projecting fibers. Then they were wiped quickly with TCE (1,1,1-trichloroethane) and rinsed with deionized water. They were stored for several weeks in a desiccator. An attempt was made to weigh the specimens before and after sputter coating to determine coating weights. However the results were inconclusive due to the extremely light weights of the coatings. The measured weights may have been influenced by varying temperature and humidity conditions in the laboratory.

TEST EQUIPMENT, PROCEDURES, AND ANALYSIS

Sputter Coater

The Torr Vac sputter deposition system which was built to Langley Research Center specifications is shown in figure 3. The system may be used for DC magnetron sputtering, RF magnetron sputtering, and RF etch. In the present study it was used in the DC magnetron configuration. It presently has three targets (source materials), one of which is aluminum. Figure 4 shows the upper portion of the vacuum chamber where the targets are located. An automatic hoist, shown in figure 3, allows the chamber cover to be raised for access to the interior. Figure 5 is a photo of the interior looking down at the planetary fixturing with six planetary plates. The planetary fixture and plates can be revolved and rotated beneath the targets to enhance uniformity of sputtered coatings. The large fan-shaped valve in the raised portion of the vacuum chamber (fig. 4) is used to contain the plasma near the target and away from the specimens until operating conditions are achieved. A louvered throttle valve between the turbomolecular pump and the vacuum chamber can be closed allowing the vacuum chamber to be backfilled with argon to provide suitable conditions for a plasma to form. The console contains process control and instrumentation as indicated in figure 3.

Sputter Conditions

Six sputter coating runs were made. For each run, ten 1-inch-diameter specimens were placed on a plate in the vacuum chamber. A thickness monitor (see next Section) was placed in the center of the plate, and arranged around it were five specimens with smooth side up and five with rough side up. During sputtering the plate was stationary, about 3 inches under the aluminum target.

All coatings were sputtered at 1 kW power. The chamber was evacuated for at least 30 minutes before sputtering, resulting in initial chamber pressures in the range of 4×10^{-6} to 1×10^{-5} torr. The chamber was backfilled with argon to a pressure of 8 microns and an arc was struck to form a plasma. The system was programmed to ramp up power to reach 1 kW in one minute after the plasma formed. At that moment the large fan valve shielding the specimens from the plasma was opened to expose the specimens to the plasma. When the desired sputtering time was achieved the valve was closed and power was turned off. The sputtering times were based on previous experience and chosen to provide a range of coating thicknesses.

Coating Thickness Determination

The coating thickness could not be measured directly on the composite material because the surface irregularities of the composite material were greater than the coating thicknesses. Therefore coating thickness monitors such as shown in figure 6 were used to obtain sputtered coating thicknesses. The sapphire wafers have very smooth surfaces which allow the height of a "step" of material that has been deposited on them to be measured with an instrument such as the Dektak 2. The coating thickness monitor was made by cutting a 7- × 12-mm piece from a sapphire wafer, placing it in a brass holder, and covering the wafer and holder with a slotted shield. The 1- × 8-mm slot in this shield was cut with an electron beam from a 1-inch-square piece of 5-mil thick spring steel. The pieces of sapphire with the sputtered aluminum step were mounted on a microscope slide for ease of handling.

The aluminum steps deposited on the sapphire wafer monitors were assumed to be the same thickness as the coatings on the specimens. However, the steps obtained were not sharply rectilinear, but were unevenly mounded with curved edges. The highest points of each step were in the center of the steps, farthest from the shield edges, and were assumed to be equal to the coating thickness on the composite specimens. The highest point measured was noted, and these six points (one from each sputter run) are plotted in figure 7 as a function of sputtering time; a straight line from the origin is faired through them. Coating thickness as a function of sputtering time was taken from this line and used in the following discussion.

Optical Properties

Solar reflectance was measured in the wavelength range of 0.3 to 2.5 micrometers with a Gier Dunkle MS-251 solar reflectometer. The source, optics, and sphere characteristics of this instrument as it was used approximate the solar spectrum. For an opaque surface the solar absorptance can be computed by subtracting the reflectance from unity. Total normal emittance of the specimens was determined from infrared reflectivity measurements made with a Gier Dunkle DB-100 infrared reflectometer in the wavelength range of 5 to 25 microns. For each surface texture and coating thickness five specimens were coated and measured and a mean with a standard deviation was calculated.

RESULTS AND DISCUSSION

Solar absorptance, α_s, and total normal thermal emittance, ϵ, were determined for six coating thicknesses and two surface textures. The results are presented in table I and figures 8 through 10.

The results indicate that sputter coating of the composite substrate dramatically lowers α_s from an undesirable value of about 0.70 to a much more desirable value of 0.16 on the smooth surface. Coating of the substrate beyond 420Å appears to have little effect on α_s, at least up to the maximum thickness of 2520Å. Although the α_s values for the rough surfaces are somewhat higher, i.e. about 0.24, this, too, is an acceptable value for space flight in a low Earth orbit (ref. 1).

Coating with aluminum lowers ϵ from 0.8 for the smooth surface of the bare composite to 0.08, an unacceptably low value for effective temperature balance in space. This problem could have been anticipated because the bare composite was a dull black and the sputtered surface is highly reflective, conductive metal. The aluminum coated rough surface, however, produces an ϵ of about 0.3 yielding a α_s/ϵ ratio of about 1, which is acceptable under some space flight conditions.

The lower α_s and ϵ values for the smooth surfaces compared with the rough surfaces can be attributed to two effects. First, sputtered coatings on rough surfaces tend to be non-uniform with thinner coatings deposited on highly sloped surfaces than on flatter surfaces. Second, with uniform rates of deposition per unit area across the planetary plate, rough specimens and smooth ones of the same diameter receive equal amounts of sputtered material. Calculations of surface area based on the roughness data shown in figure 2 indicate that the rough specimens present about 1.3 times as much surface area as the smooth ones. The coating will be thinner on rough substrates than on smooth ones (reference 17); and because of this the affect of the coating will be reduced.

The α_s of aluminum coating was expected to decrease to a minimum at coating opacity and then to level off. By and large, this is what was observed. For the rough surfaces the minimum occurred at around 1000Å (see Figure 8). For the smooth surfaces the minimum occurred at about 400Å. At greater coating thicknesses, solar absorptances generally remained constant although there is the hint of slight increases out to about 2520Å. Oxidation of the aluminum during sputtering may have caused these slight increases since electron dispersive X-ray analysis of the coatings show the presence of oxygen. The decrease of emittance with increasing coating thickness (Figure 9) was expected. The ratio of α_s/ϵ is approximately 1 for the rough surfaces and increases slightly with coating thickness (Figure 10). The α_s/ϵ ratio for the smooth surfaces rises from 2 to 4 over the coating thickness range of 420Å to 2520Å. These coatings (whether applied to smooth or rough surfaces) could be of limited use as thermal control coatings for composites in large space structures in low Earth orbits.

SUMMARY OF RESULTS

The following observations were made in the course of this investigation on sputtered aluminum coatings on graphite/epoxy composite materials:

1) Surface texture of the initial uncoated specimen influences the optical properties of the coated surface.
2) Solar absorptance and emittance were lower for the smooth specimens than for the rough ones.
3) The ratio of α_s/ϵ was higher for the smooth specimens, increasing from 2 to 4 over the range of coating thicknesses applied, than for the rough ones, which had a ratio of about 1.
4) A coating of just 420Å thickness reduces solar absorptance from 0.7 for the uncoated composite to 0.3 for a rough surface and 0.2 for a smooth surface.
5) Total normal emittance values decreased with increasing coating thickness.
6) After reaching a minimum α_s at around 1000Å for the rough surfaces and 400Å for the smooth surfaces, α_s exhibited slight increases up to coating thicknesses of 2520Å, possibly due to oxide contamination of the aluminum.

REFERENCES

1. Foss, R. A.: Evaluation of Graphite/Epoxy Tubular Space Station Structures. AIAA 24th Aerospace Science Meeting. Reno, NV. January 1986.
2. Adams, D. S.; Bowles, D. E.; and Herakovich, C. T.: Thermally Induced Transverse Cracking in Graphite/Epoxy Cross-Ply Laminates. Journal of Reinforced Plastics and Composites, July 1986.
3. Hyer, M. W.; Cohen, D.; Cooper, D.; and Tompkins, S. S.: Temperature and Deformation Data from the Asymmetric Heating of Cross-Ply Composite Tubes. Journal of Composite Technology and Research. Vol. 9. No. 1, 1987, pp. 14-20.
4. Tompkins, S. S.; Bowles, D. E.; Slemp, W. S.; and Teichman, L. A.: Response of Composite Materials to the Space Station Orbit Environment. Space Station Symposium (AIAA/NASA), Williamsburg, VA. April 21-22, 1988.
5. Robinson, Raymond S.: Ion Beam Microtexturing and Enhanced Surface Diffusion. NASA CR-167948, February 1982.
6. Adams, R. O.; Nordin, C. W.; and Masterson, K. D.: Aluminum-Silver Alloy Films for Solar Reflectors. Presented at the International Conference on Metallic Coatings, San Diego, CA. Rockwell International Corp. Rep. 3021, 21 April 1980.
7. Craighead, H. G.; Howard, R. E.; Sweeney, J. E.; and Tennant, D. M.: Textured Surfaces: Optical Storage and Other Applications. J. Vac. Sci. Technol., 20(3), March 1982.
8. Cuomo, J. J.; Ziegler, J. F.; and Woodall, J. M.: A New Concept for Solar Energy Thermal Conversion. Applied Physics Letters, Vol. 26, No. 10, 15 May 1975.

9. Kanayama, Kimio: Total Emittance and Monochromatic Emittance of Metals and Nonmetals. Journal of Heat Transfer-Japanese Research, Volume 2, July-September 1973.

10. Loefving, Sten: Hemispherical Emittance of Rough Metal Surfaces. Appl. Phys. Lett. 36(8), 15 April 1980.

11. Gittleman, J. I.; Sichel, E. K., Lehmann, H. W.; and Widmer, R.: Textured Silicon: A Selective Absorber for Thermal Conversion. Appl. Phys. Lett. 35(10), 15 November 1979.

12. Siegfried, R. G.; and Birkebak, R. C.: A Comparative Study of the Total Directional Emittance of a Dielectric and a Metal as a Function of Surface Roughness. Presented at the AIAA 3rd Thermophysics Conference, Los Angeles, CA, June 24-26, 1968. AIAA No. 68-778.

13. Hudson, Wayne R.: Ion-Beam Texturing of Materials. NASA Tech Briefs, p. 374, Fall 1978.

14. Harding, G. L.; and Lake, M. R.: Sputter Etched Metal Solar Selective Absorbing Surfaces for High Temperature Thermal Collectors. Solar Energy Materials, Vol. 5, No. 4, 1981.

15. Carlsson, Leif A.; and Pipes, R. Byron: Experimental Characterization of Advanced Composite Materials. Prentice-Hall, Inc., 1987.

16. Douglas Material Specification 2163E, 21 January 1983, McDonnell Douglas Corp.

17. Esmore, L. H.; Mourant, B. L.; and Reeves, F. M.: The Measurement of the Thickness of Electroplated Coatings on Rough Surfaces. Materials Research Laboratories, Maribynong Victoria. Report 610, November 1974.

Table I. Solar Absorptance, Total Normal Emittance, and the Ratio α_s/ϵ

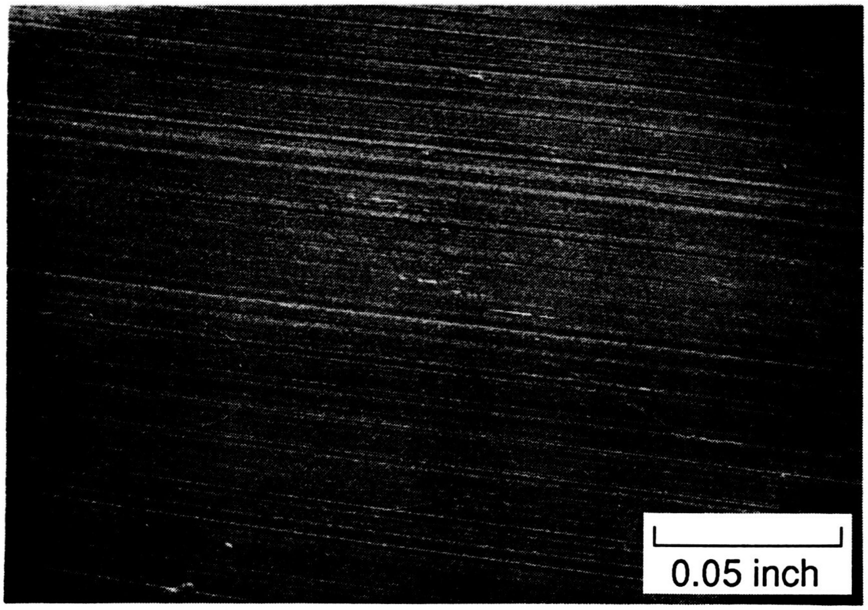
	Smooth			Rough		
Coating thickness	α_s	ϵ	α_s/ϵ	α_s	ϵ	α_s/ϵ
Uncoated	.694	.806	.861	.703	.814	.864
	.695	.802	.867	.700	.821	.853
	.692	.806	.859	.708	.811	.873
	$\bar{x}$ = .694	.805	.862	.704	.815	.863
	s_x = .002	.002		.004	.005	
420Å	.156	.075	2.080	.271	.271	1.000
	.168	.083	2.024	.293	.300	.977
	.165	.088	1.875	.302	.352	.858
	.168	.100	1.680	.298	.324	.920
	.165	.091	1.813	.266	.256	1.039
	$\bar{x}$ = .164	.087	1.894	.286	.301	.959
	s_x = .005	.009		.016	.039	
840Å	.160	.061	2.623	.242	.230	1.052
	.158	.073	2.164	.251	.247	1.016
	.158	.063	2.508	.244	.243	1.004
	.174	.087	2.000	.246	.218	1.128
	.197	.113	1.743	.239	.238	1.004
	$\bar{x}$ = .169	.079	2.208	.244	.235	1.041
	s_x = .017	.021		.005	.012	
1260Å	.166	.070	2.371	.255	.269	.948
	.148	.059	2.508	.243	.253	.960
	.172	.087	1.977	.221	.207	1.078
	.150	.046	3.261	.222	.205	1.083
	.150	.057	2.632	.219	.209	1.048
	$\bar{x}$ = .157	.064	2.550	.232	.229	1.023
	s_x = .011	.016		.016	.030	
1680Å	.164	.045	3.644	.258	.224	1.152
	.195	.065	3.000	.243	.227	1.070
	.179	.073	2.452	.261	.237	1.101
	.188	.052	3.615	.256	.216	1.185
	.171	.062	2.758	.243	.223	1.090
	$\bar{x}$ = .179	.059	3.094	.252	.225	1.120
	s_x = .013	.011		.009	.008	

Table I. Concluded.

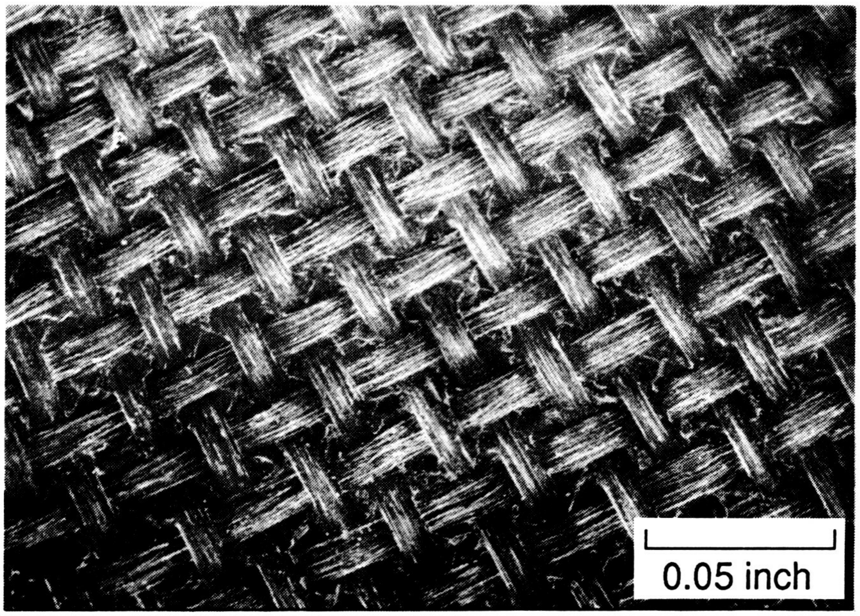
	Smooth			Rough		
Coating thickness	α_s	ϵ	α_s/ϵ	α_s	ϵ	α_s/ϵ
2100Å	.201	.044	4.568	.254	.180	1.411
	.168	.046	3.652	.261	.201	1.299
	.160	.035	4.571	.262	.227	1.154
	.156	.050	3.120	.249	.197	1.264
	.178	.067	2.657	.325	.195	1.667
	$\bar{x}$ = .173	.048	3.714	.270	.200	1.359
	s_x = .018	.012		.031	.017	
2520Å	.194	.050	3.880	.262	.186	1.409
	.206	.051	4.039	.277	.197	1.406
	.183	.043	4.256	.248	.176	1.409
	.178	.039	4.564	.267	.183	1.459
	.204	.045	4.533	.305	.223	1.368
	$\bar{x}$ = .193	.046	4.254	.272	.193	1.410
	s_x = .012	.005		.021	.018	

$$\bar{x} = \text{mean} = \frac{1}{n} \sum_{i=1}^{n} x_i$$

$$s_x = \text{standard deviation} = \sqrt{\frac{\Sigma x_i^2 - n\bar{x}^2}{n - 1}}$$

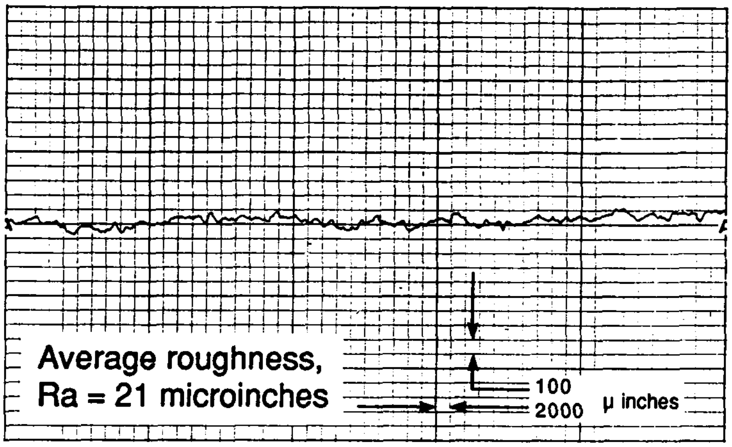


(a) Smooth surface.

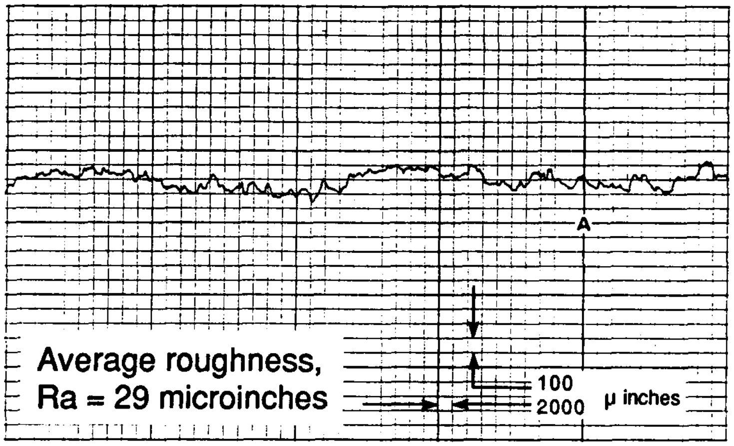


(b) Rough surface.

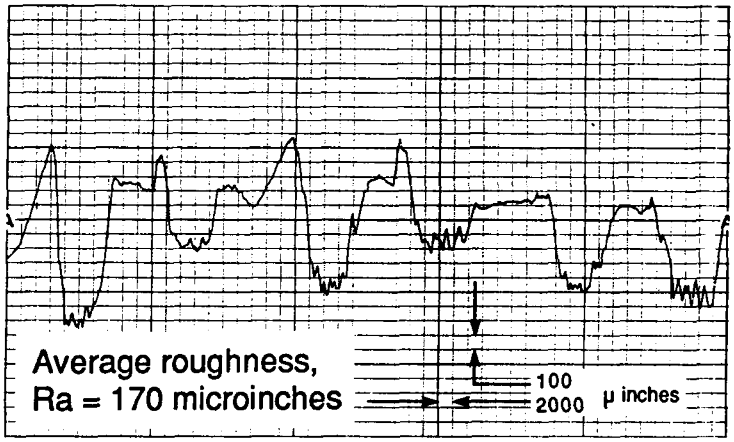
Figure 1.- SEM images of composite surfaces.



(a) Smooth side, along striations



(b) Smooth side, across striations



(c) Rough side

Figure 2.- Surface analyzer traces of composite surfaces.

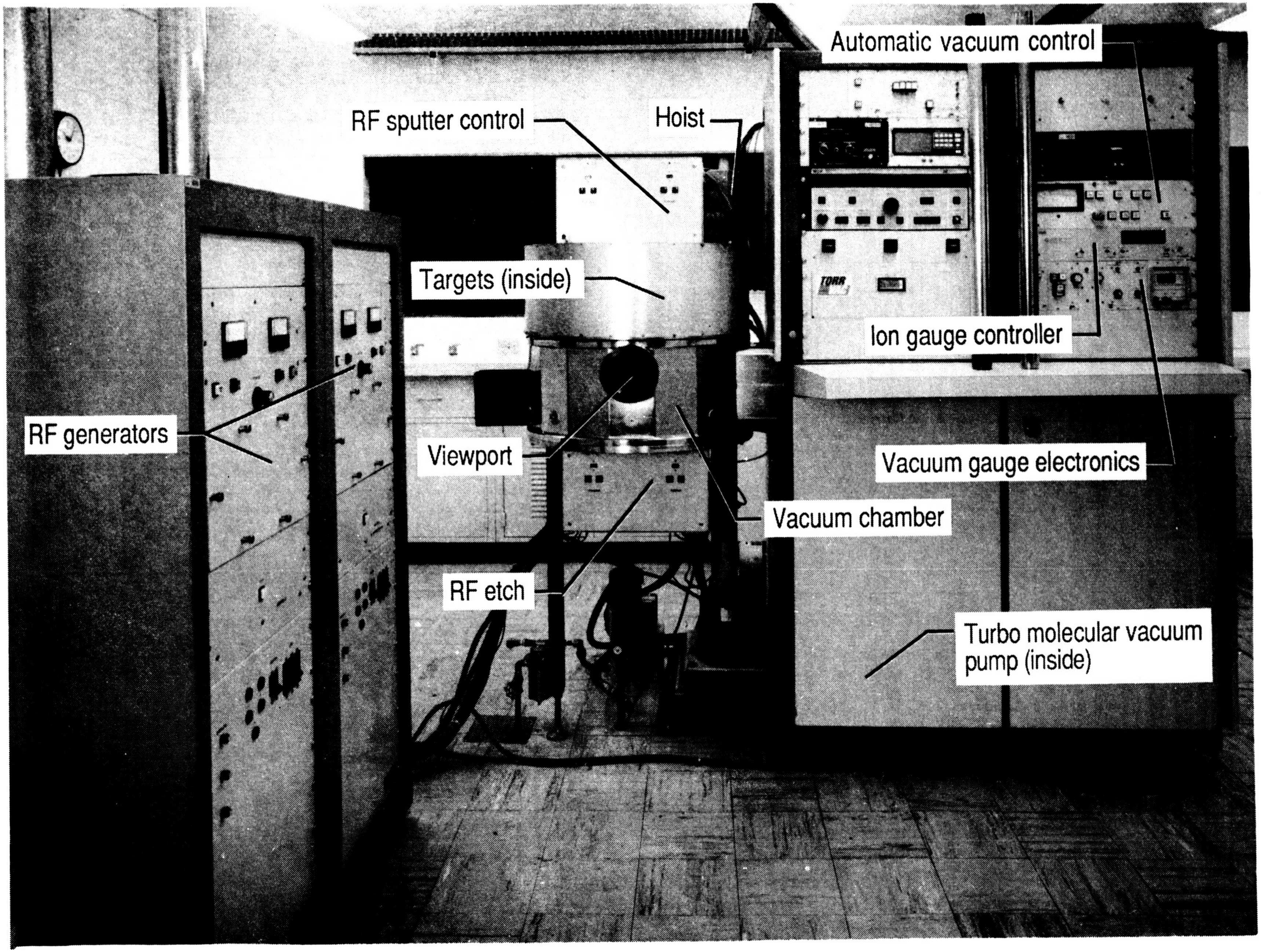
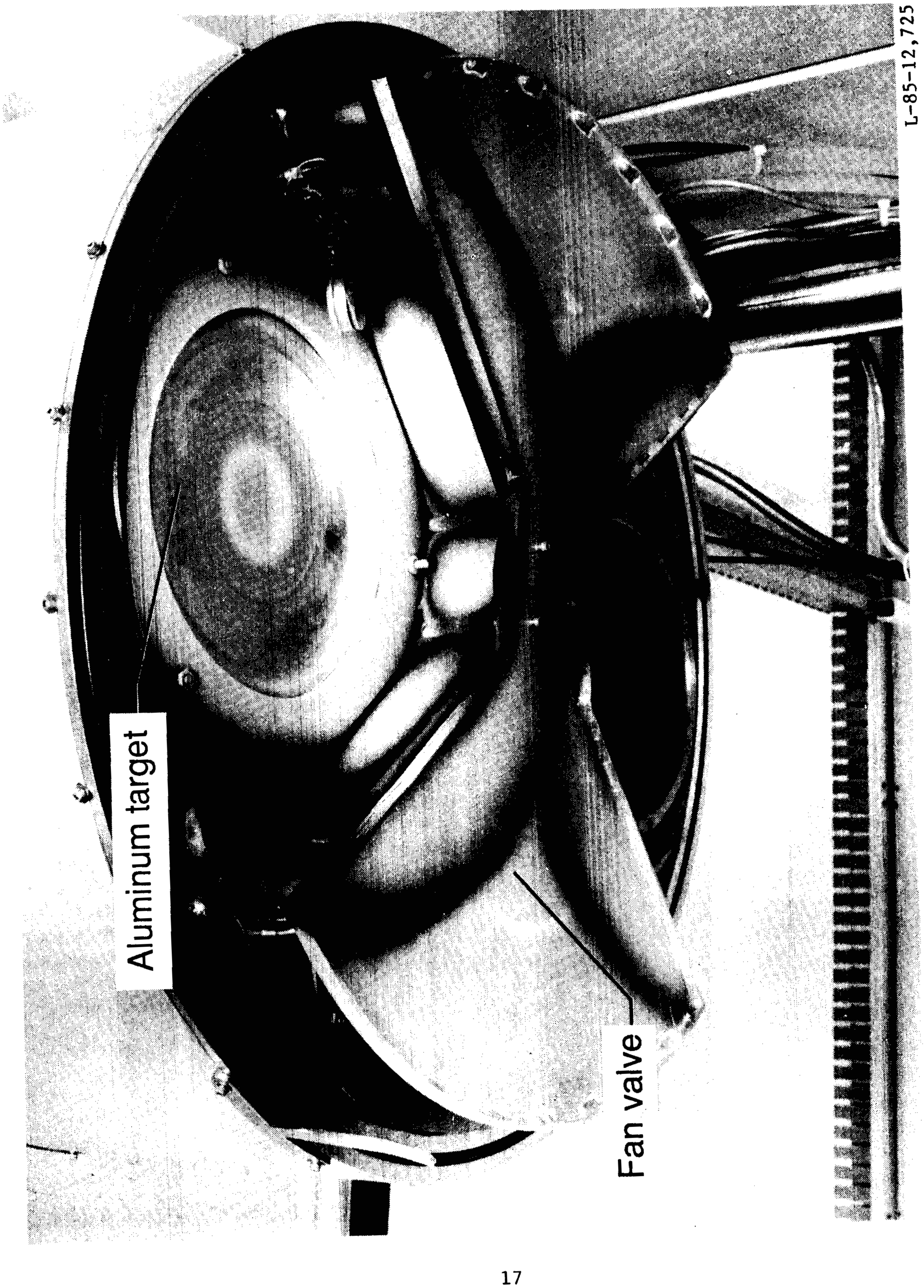


Figure 3.- Sputter deposition system.

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH



L-85-12,725

Figure 4.- Upper portion of the sputter coating chamber.

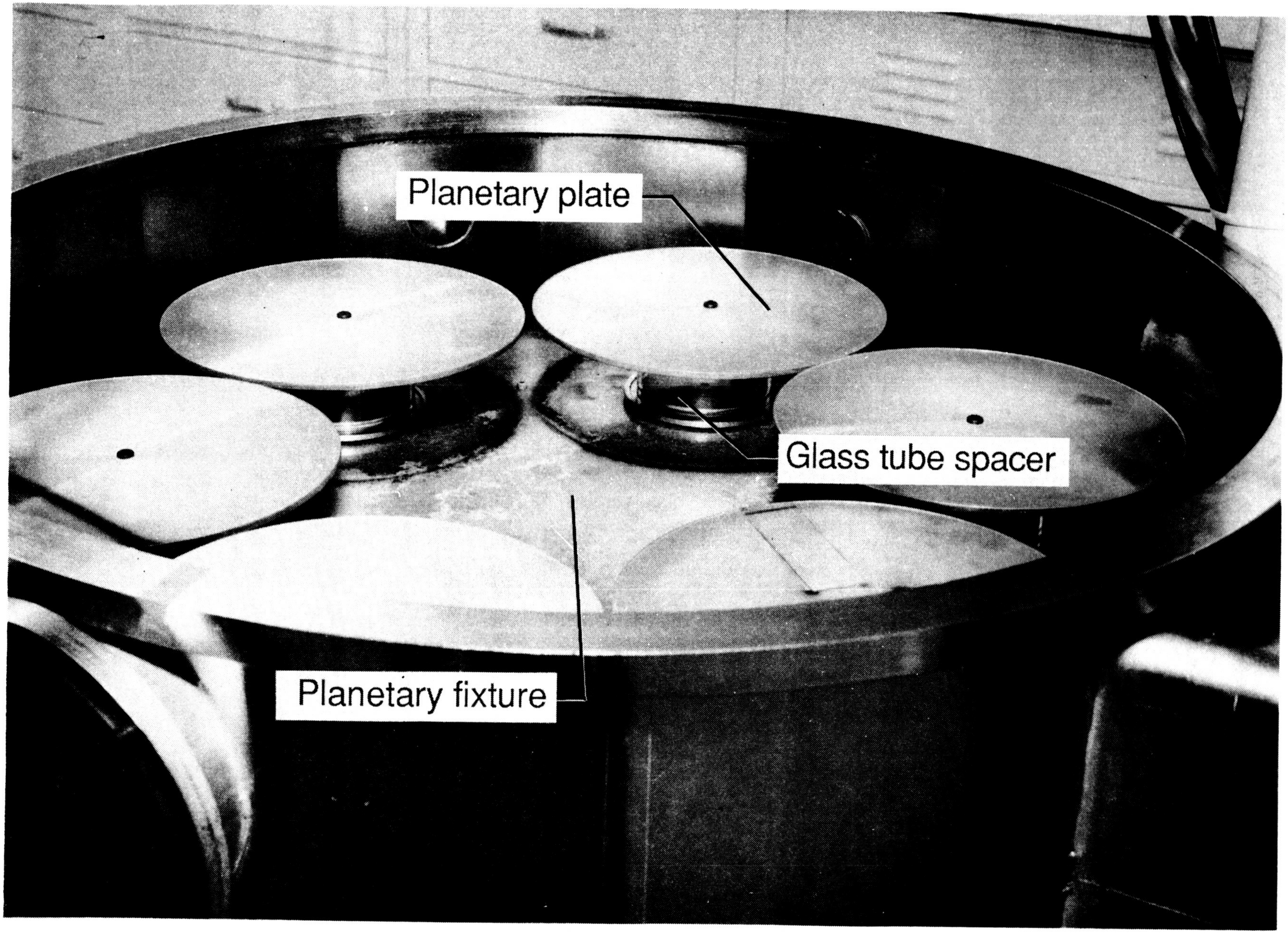


Figure 5.- Lower portion of the sputter coating chamber.

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

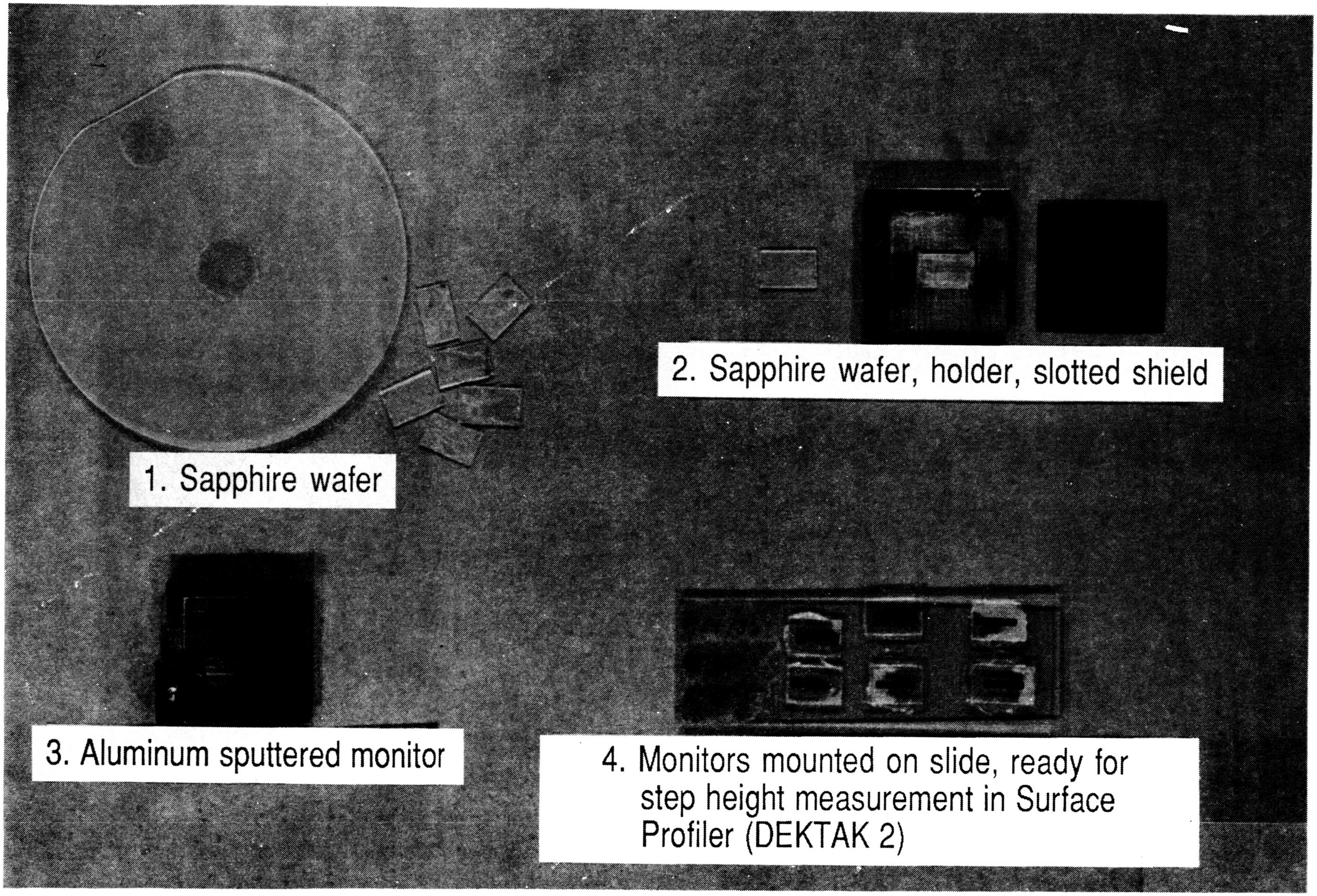


Figure 6.- Sputtered coating thickness monitor.

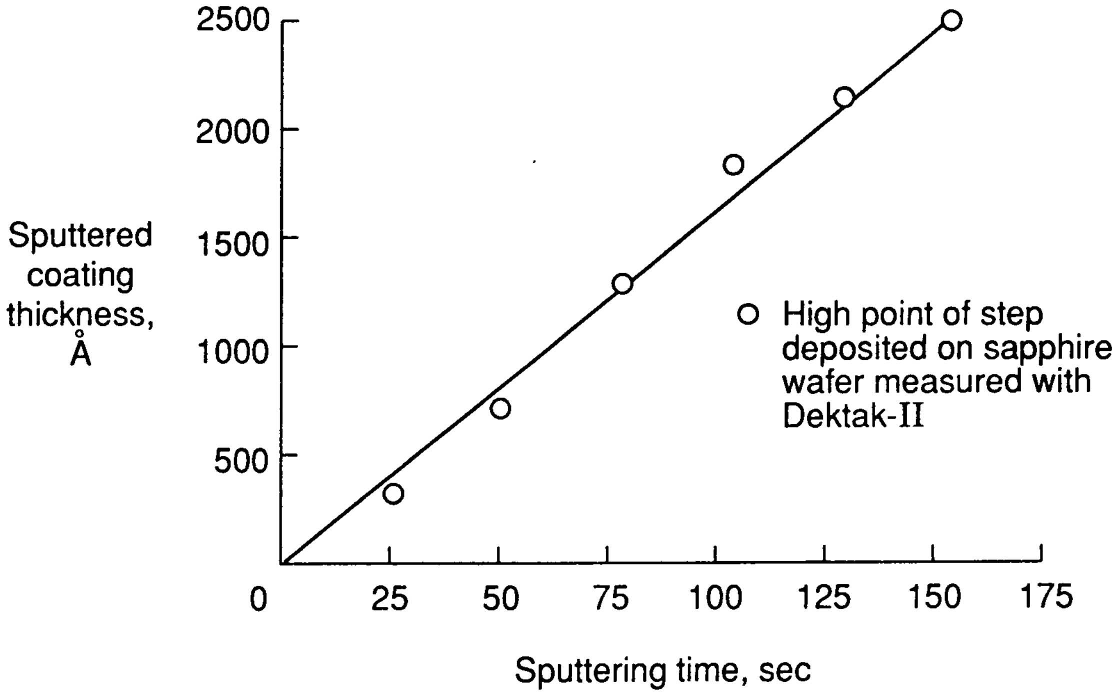


Figure 7.- Aluminum sputtered coating thickness as a function of sputtering time.

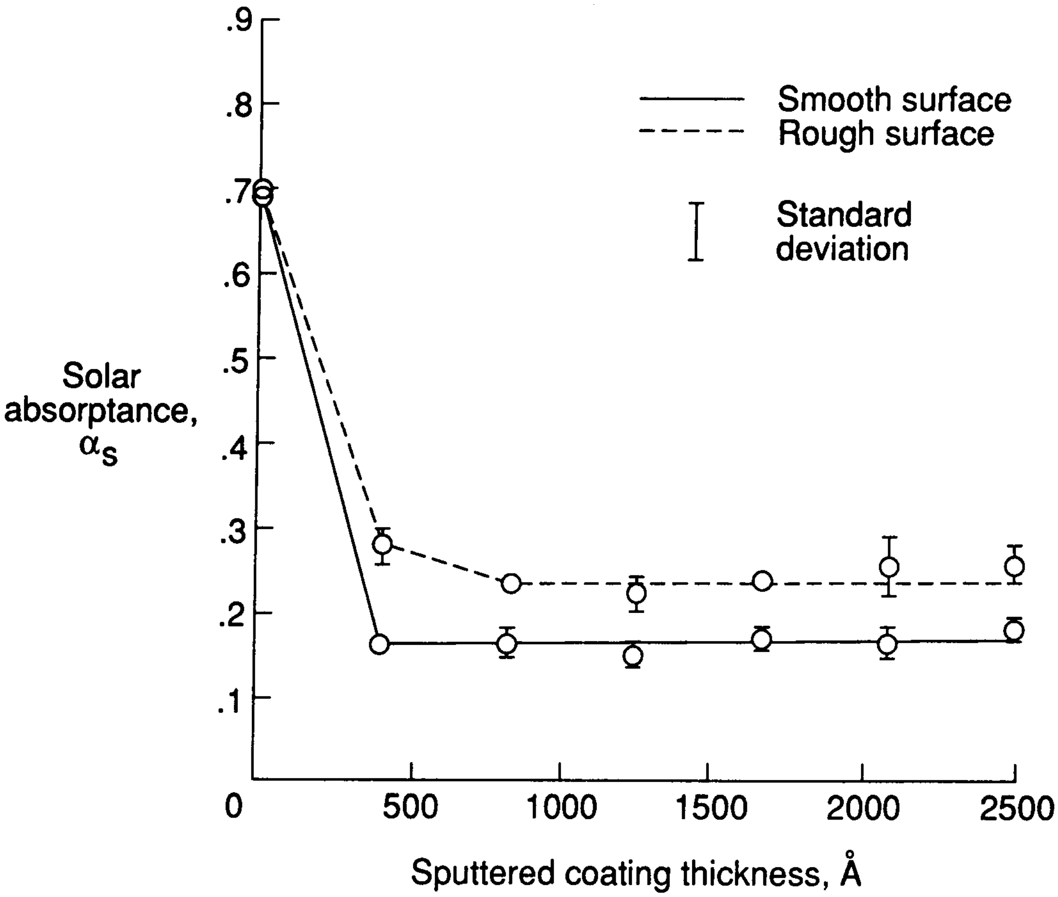


Figure 8.- Solar absorptance of sputtered aluminum on T300/5209 as a function of sputtered coating thickness.

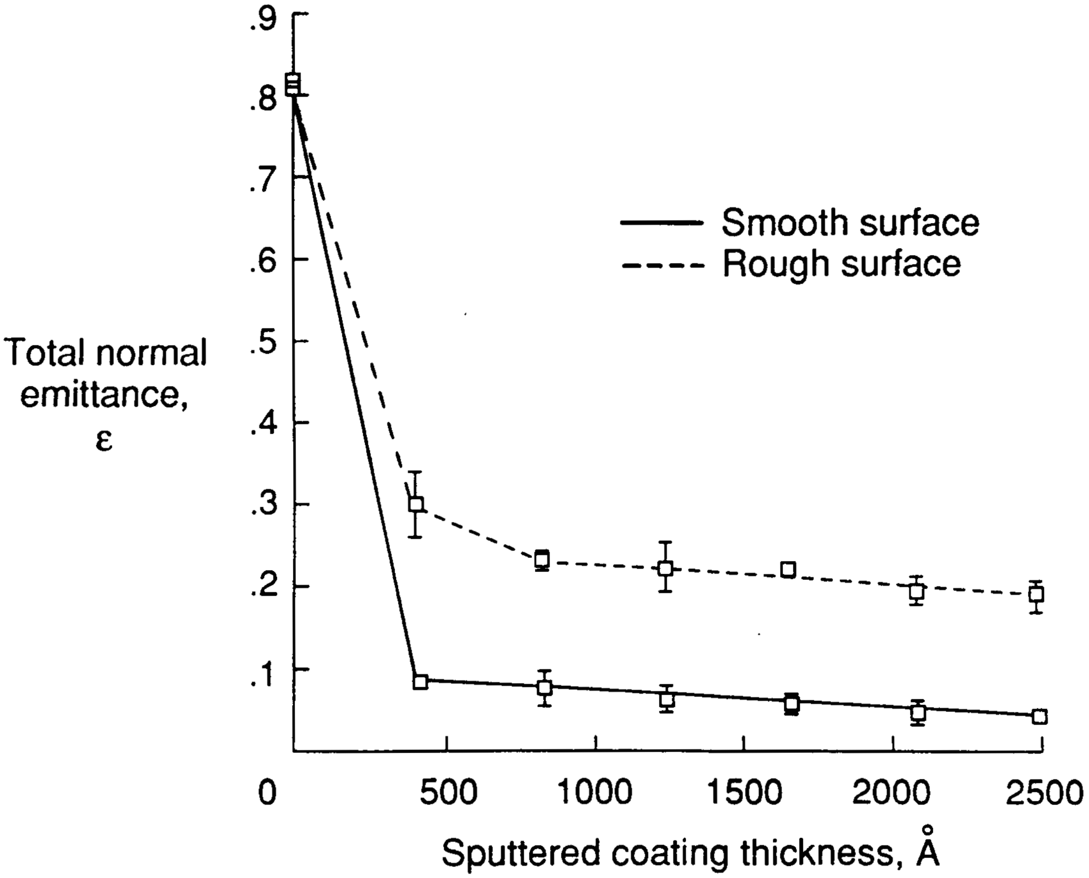


Figure 9.- Total normal emittance of sputtered aluminum on T300/5209 as a function of sputtered coating thickness.

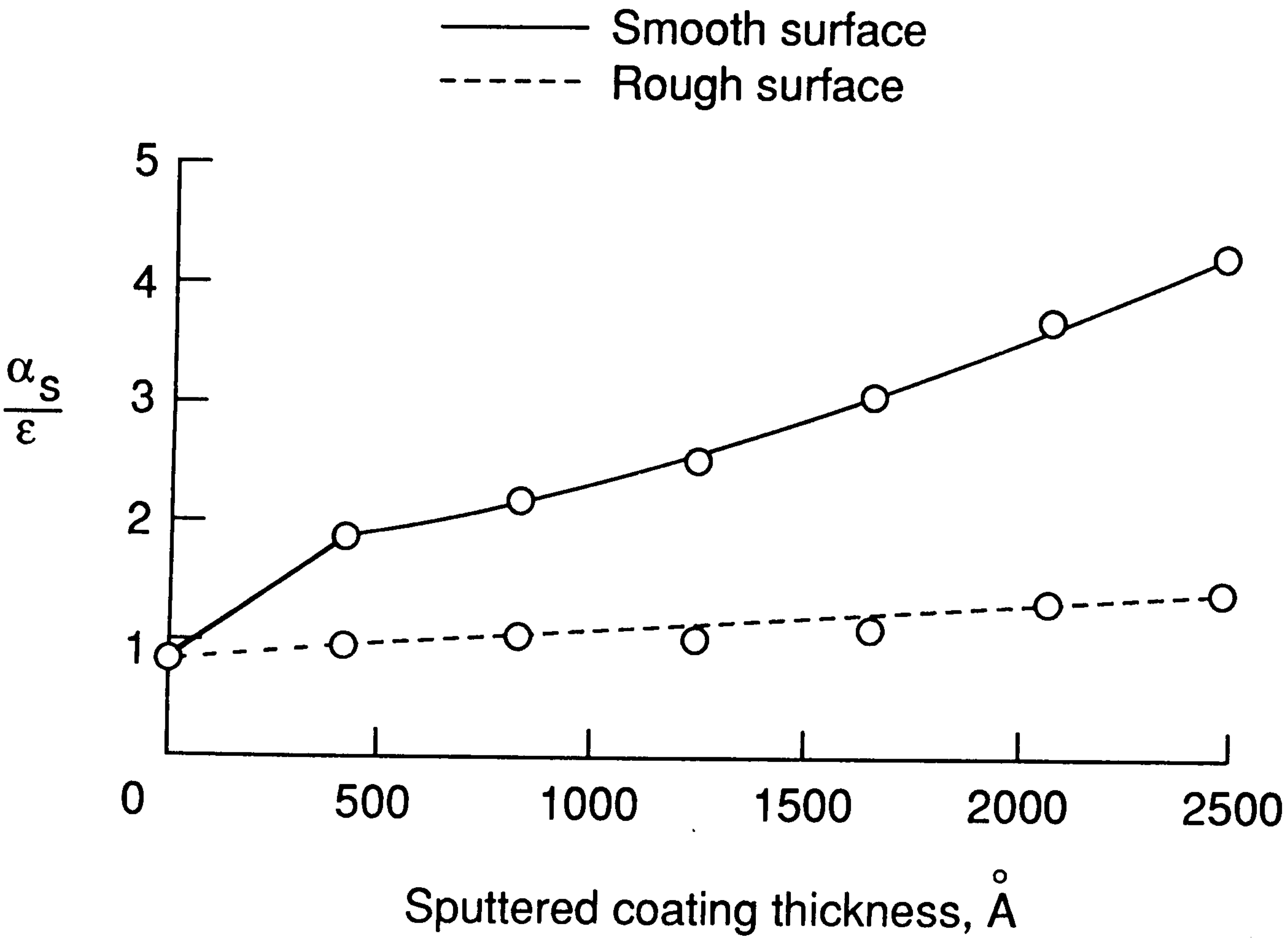


Figure 10.- The ratio a_s/e of sputtered aluminum as a function of coating thickness on T300/5209.

National Aeronautics and Space Administration

Report Documentation Page

1. Report No. NASA TM-101620	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle OPTICAL PROPERTIES OF SPUTTERED ALUMINUM ON GRAPHITE/EPOXY COMPOSITE MATERIAL	5. Report Date August 1989
	6. Performing Organization Code
7. Author(s) William G. Witte, Jr. Louis A. Teichman	8. Performing Organization Report No.
	10. Work Unit No. 506-43-21-04
9. Performing Organization Name and Address NASA Langley Research Center Hampton, VA 23665-5225	11. Contract or Grant No.
	13. Type of Report and Period Covered Technical Memorandum
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, DC 20546-0001	14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

Solar absorptance, emittance, and coating thickness were measured for a range of coating thicknesses from about 400Å to 2500Å. The coatings were sputtered from an aluminum target onto 1-inch-diameter substrates of T300/5209 graphite/epoxy composite material with two different surface textures. Solar absorptance and emittance values for the specimens with the smooth surface finish were lower than those for the specimens with the rough surface finish. The ratio of solar absorptance to emittance was higher for the smooth specimens, increasing from 2 to 4 over the coating thickness range, than for the rough ones, which had a constant ratio of about 1. The solar absorptance and emittance values were dependent on the thickness of the sputtered coating.

17. Key Words (Suggested by Author(s)) Sputtering Solar Absorptance Emittance Graphite/Epoxy Aluminum	18. Distribution Statement Unclassified - Unlimited Subject Category - 37

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price
Unclassified	Unclassified	24	A03