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#### Collection and Review of Metals Data Obtained From LDEF Experiment Specimens and Support Hardware\*

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#### INTRODUCTION

LDEF greatly extended the range of data available for metals exposed to the low-Earth-orbital environment. The effects of low-Earth-orbital exposure on metals include meteoroid and debris impacts, solar ultraviolet radiation, thermal cycling, cosmic rays, solar particles and surface oxidation and contamination. This paper is limited to changes in surface composition and texture caused by oxidation and contamination. Surface property changes afford a means to study the environments (oxidation and contamination) as well as in-space stability of metal surfaces.

In this paper we will compare thermal-optical properties for bare aluminum and anodized aluminum clamps flown on LDEF. We will also show that the silicon observed on the LDEF tray clamps and tray clamp bolt heads is not necessarily evidence of silicon contamination of LDEF from the Shuttle. The paper concludes with a listing of LDEF reports that have been published thus far that contain significant findings concerning metals.

### BARE ALUMINUM AND ANODIZED ALUMINUM CLAMPS

The retaining clamps on LDEF Experiment Trays C9 and C3 offer an opportunity to compare the behavior of bare and chromic acid anodized (CAA) aluminum when exposed to space in low-Earth-orbit (LEO). Figure 1 shows a photograph of Tray C9. The four corner clamps that hold this tray in place on the vehicle are bare 6061-T6 aluminum. The remaining four clamps on Tray C9 are anodized aluminum. Figure 2 shows a close-up of the bare aluminum Clamp C09-3 on Tray C9 and is adjacent to the CAA Clamp C08-5 on Tray C8. Both Clamp C09-3 and Clamp C08-5 are bolted to Longeron 8-9 at an angle of 23 degrees to ram. A striking difference in the appearance of the anodized and bare aluminum surfaces is shown in the photographs.

Tray clamps on LDEF were protected by their location from possible line-of-sight sources of contamination from the LDEF vehicle itself as shown in Figure 3. It is assumed that very little contamination from the vehicle reached the clamp surfaces. Two bare aluminum clamps, (C09-7, leading edge and C03-5, trailing edge) and two CAA clamps (C09-2, leading edge and C03-6, trailing edge) were selected for laboratory testing and comparison of thermal-optical properties. The locations of the four clamps included in the comparison of bare and anodized aluminum surfaces are shown in Figure 4. Solar radiation and atomic oxygen exposure data for these clamps are shown on Table 1.

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Solar absorptance and thermal emittance are shown on Table 1 for the thermaloptical properties comparison. Data for flight clamps are from post-flight measurements. Data for control clamp #4 were taken after the clamp was removed from storage. Solar absorptance was measured in accordance with ASTM E903-82 and ASTM E424-17. Thermal emittance was measured in accordance with ASTM E408-71.

Flight exposure caused little change in the thermal-optical properties of either bare or CAA clamp surfaces (Table 1). A bare ground control clamp was not available. However, thermal-optical properties for the exposed surfaces of bare flight clamps may be compared with those of unexposed surfaces (back surfaces) of the same clamps. Thermaloptical properties of exposed surfaces of CAA flight clamps may be compared with those of the ground control clamp.

The thermal-optical properties of bare clamp surfaces and CAA clamp surfaces differ significantly. Average solar absorptance for bare flight clamp surfaces (leading edge and trailing edge) is 210 percent that of CAA flight clamp surfaces. Average thermal emittance for bare flight clamp surfaces (leading edge and trailing edge) is 45 percent that of CAA flight clamp surfaces.

Figures 5 through 9 give Auger electron spectroscopy profiles (AES) for principal elements on the surfaces of the clamps. The conversion of sputter time to depth is estimated at approximately 120 Angstroms/minute. Figures 5 and 6 suggest that magnesium near the surface of bare aluminum plate is higher than the bulk average value deeper in the material. This variation in composition at the surface of metal alloys is caused by the manufacturing process. This surface composition change with depth is not evident on CAA aluminum because the surface is etched before anodizing and the anodizing process converts aluminum to aluminum oxide.

Figures 5 and 6 show that based on post-flight determinations the depth at which magnesium and aluminum concentrations are equal is approximately 890 Angstroms (7.4 minutes sputter time) for the leading-edge bare clamp C09-7 and approximately 1150 Angstroms (9.6 minutes sputter time) for the trailing-edge bare clamp C03-5. The difference is attributed to the removal of carbon by atomic oxygen for the leading-edge clamp.

Figures 5 and 6 show lower carbon concentrations for Clamp C09-7 than for Clamp C03-5 at depths greater than 200 Angstroms (2 minutes sputter time). Clamp C03-5 was shielded from exposure to atomic oxygen. However, surface concentrations of carbon obtained by post-flight measurements are the same for both bare clamps, C09-7 (leading edge) and C03-5 (trailing edge). It is likely that the carbon content of the clamp surfaces increased due to exposure to the post-flight, ambient ground atmosphere.

Figure 7 shows ESCA atomic concentrations for a CAA treated control clamp. The control clamps were stored in a laboratory at NASA-Langley during the LDEF mission. Figures 8 and 9 when compared with Figure 7 for the control clamp surface show that the CAA treated clamp surfaces were unaffected by exposure to the LEO environment whether exposed on leading or trailing surfaces of LDEF.

#### ESCA DETERMINATIONS ON BOLT-HEAD SURFACES

Figure 3 shows that the tops of the stainless steel bolts that held the clamps in place were shielded from possible contamination from sources on-board LDEF. This positioning of the bolt heads makes them useful for studying possible contamination of LDEF surfaces by the Shuttle. The location of the bolts also prevented them from being shielded from atomic oxygen and solar ultraviolet radiation exposures. Likewise, the bolt head tops were not exposed to either the atomic oxygen flux or solar radiation reflected from other LDEF surfaces. Thus, prediction of oxidation and radiation environments for the bolt head tops is simplified.

ESCA determinations were made on the tops of the clamp bolt heads. Figure 10 shows that the atomic concentration of silicon increases everywhere around the LDEF vehicle relative to the control bolts. However, no trend in the absolute amount of silicon on the bolt heads is necessarily indicated by Figure 10. The alloy from which the bolts are manufactured has a small concentration of silicon. The apparent increase in silicon atomic concentration may be caused by the removal of carbon atoms by atomic oxygen. Carbon concentrations tend to decrease with increasing atomic oxygen exposure.

Figure 11 is a plot of the ratio of silicon-to-iron concentrations for locations around the vehicle. If it is assumed that the quantity of iron present on the bolt-head surfaces is constant (unaffected by atomic oxygen) then the amount of silicon on the bolts would vary with the silicon-to-iron ratio as plotted in Figure 11.

If LDEF were being contaminated by silicon from the Shuttle during deployment or recovery then a definite trend in relative silicon-to-iron composition with position on the vehicle would be expected. No consistent trend is seen in this ratio. The variation in the silicon-to-iron ratio shown by the plot may be explained by random errors in the analytical procedure and preflight condition of the bolt surfaces. These ESCA data do not necessarily support the premise of silicon contamination of LDEF from outgassing from the Shuttle.

Figure 12 shows both atomic oxygen fluence and carbon atomic concentrations on bolt heads as a function of position on LDEF. Figure 12 shows that the reduction in carbon atomic concentration on the stainless steel bolt heads is a sensitive indicator of atomic oxygen exposure, not withstanding the universal availability of carbon in the atmosphere that might change the surface carbon concentration following the LDEF flight. Note that this finding for stainless steel surfaces is opposite to the finding for bare aluminum clamp surfaces discussed above. For bare aluminum surfaces, the concentration of carbon following flight tended to be independent of atomic oxygen exposure. This effect was attributed to post-flight reaction of the aluminum surfaces with carbon in the ground atmosphere.

#### **AES PROFILES ON BOLT-HEAD SURFACES**

It was decided that a comparison of Auger electron spectroscopy profiles for control bolts and flight bolts would be useful in explaining the surface composition changes caused by the LEO environment. Figure 13 shows the locations on LDEF of six flight bolts chosen for the comparison. Figure 14 shows the average silicon-to-iron ratios obtained for four unexposed control bolts. The range of the ratio at any given depth is quite wide, but the average values form a smooth curve. Five of the flight bolts (E9-4c, D7-7a, A6-6c, D2-4c, and H11-7a) were exposed to significant amounts of atomic oxygen, as shown on Table 2. Figure 15 shows silicon-toiron ratios for each of these bolts compared with the average silicon-to-iron ratio for the four control bolts shifted by 45 Angstroms. The surfaces of the flight bolts show less silicon relative to iron than do the control bolts. However, the silicon-to-iron ratios for the control bolts and flight exposed bolts compare when the curves are shifted by 45 Angstroms relative to each other as shown in Figure 15. It is suggested that possibly a thin layer (45 Angstroms) of silicone material on the surface of the flight bolts may have been removed by oxidation during the mission.

The sixth flight bolt, D5-8c, received only a negligible amount of atomic oxygen. Figure 16 shows the silicon-to-iron concentration ratio at two locations for flight bolt D5-8c as a function of depth by AES profile analysis. Silicon-to-iron atomic concentration ratios for bolt D5-8c bracket the control bolt average values. This result is consistent with the fact that the head of this bolt (D5-8c) was not exposed to significant atomic oxygen during the flight. It ended the flight in a condition comparable to the average condition of the control bolts which were not exposed.

The data developed from the AES profiles on the stainless steel bolt-head surfaces does not support the premise of silicon contamination of LDEF by the Shuttle.

#### SUMMARY OF OBSERVATIONS

Table 3 lists papers published to date that contain information concerning metals flown on LDEF. The table includes papers presented at the First Post-Retrieval Symposium, the Materials Workshop '91, and the Second Post-Retrieval Symposium. A paper presented at the Third Post-Retrieval Symposium, and three other papers are also included. Additional information will become available when papers presented at the LDEF Materials Results For Spacecraft Applications Conference and the Third Post-Retrieval Symposium are published.

A number of observations concerning the effects of the LEO environment on metals have been made and confirmed by investigators reporting thus far. Some of the principal findings, including those discussed herein, can be summarized as follows:

- (1) Metals are highly variable in their response to the LEO environment.
- (2) Gold and platinum are nonreactive.
- (3) Osmium, which forms a volatile oxide, is rapidly eroded.
- (4) Silver, which forms a nonprotective oxide, is rapidly eroded.
- (5) Other metals (Al, Cu, Ga, Ge, Ir, Mo, Ni, Ti, and Sn) show some level of reaction unless protected.
- (6) Contamination is a major contributor to exposure effects on metal surfaces.

- (7) Absorptance is significantly greater for bare aluminum than for CAA aluminum. Emittance is significantly less for bare aluminum than for CAA aluminum.
- (8) The surface properties of both bare aluminum and CAA aluminum are little changed by exposure to LEO environmental conditions.
- (9) Magnesium in aluminum alloys is preferentially oxidized relative to aluminum.
- (10) An oxide coating forms on exposure of copper to atomic oxygen that impedes further oxidation.
- (11) The oxide coating adversely affects the optical properties of copper.
- (12) Copper without surface protection may be used for extended periods of time in applications where thermal management and optical performance requirements are not critical.

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SAMPLE	EXPOSURE	SURFACE TREATMENT	AVERAGE SOLAR ABSORPTANCE	AVERAGE THERMAL EMITTANCE	FIGURE NUMBER
C03-5 Trailing Edge	Back Surface No Direct Exposure	Bare	0.71	0.13	
C03-5 Trailing Edge	Longeron 3-4 2.66E+03 AO/cm <sup>2</sup> 11,000 ESH Solar	Barə	0.74	0.08	5
C09-7 Leading Edge	Back Surface No Direct Exposure	Bare	0.72	0.09	
C09-7 Leading Edge	Longeron 9-10 9.02E+21 AO/cm <sup>2</sup> 11,200 ESH Solar	Bare	0.69	0.06	6
Control #4	Ground Control	CAA	0.32	0.18	7
C03-6 Trailing Edge	Longeron 3-4 2.66E+03 AO/cm <sup>2</sup> 11,000 ESH Solar	CAA	0.35	0.14	8
C09-2 Leading Edge	Longeron 8-9 8.36E+21 AO/cm <sup>2</sup> 10,500 ESH Solar	CAA	0.33	0.17	9

## TABLE 1. THERMAL-OPTICAL PROPERTIES OF BARE AND ANODIZED ALUMINUM CLAMPS

Note: A bare ground control clamp is not available.

# TABLE 2. FLIGHT BOLTS USED IN THE AUGER ELECTRON SPECTROSCOPYPROFILE STUDY OF SILICON CONCENTRATION

BOLT NUMBER	ANGLE degrees	AO FLUENCE atoms/cm <sup>2</sup>	NUMBER OF PROFILES
A6-6c	83	1.16E+21	1
D2-4c	142	1.54 <b>E</b> +17	2
D5-8c	128	9.60E+12	2
D7-7a	53	5.45E+21	1
E9-4c	8	8.99E+21	1
H11-7a	89	4.59E+20	1

## Table 3. Metals Related Information

LDEF First Post-Retrieval Symposium Reference	Metals Related Information	Experiment
MEASUREMENTS OF EROSION CHARACTERISTICS FOR METAL AND POLYMER SURFACES USING PROFILOMETRY Ligia C. Christl, John C. Gregory and Palmer N. Peters p723	Roughness, crosion depths and material growth. 128 solid surfaces, including: iridium, gold, silver, copper, osmium, platinum, tungsten, aluminum, and molybdenum.	A0114
EFFECT ON LDEF EXPOSED COPPER FILM AND BULK Palmer N. Peters, John C. Gregory, Ligia C. Christl and Ganesh N. Raikar p755	Discussion of methods for characterizing exposed surfaces. Atomic concentrations of elements on surfaces of exposed copper film and bulk copper.	A0114
LDEF EXPERIMENT A0034: ATOMIC OXYGEN STIMULATED OUTGASSING Roger C. Linton, Rachel R. Kamenetzky, John M. Reynolds and Charles L. Burris p763	Optical degradation of the gold, silver, and osmium mirrors.	A0034
PRELIMINARY RESULTS FOR LDEF/HEPP THERMAL CONTROL SAMPLES Lonny Kauder p797	Atomic oxygen resistant vapor deposited aluminum coatings for Kapton.	
INITIAL MATERIALS EVALUATION OF THE THERMAL CONTROL SURFACES EXPERIMENT (S0069) Donald R. Wilkes, M. John Brown, Leigh L. Hummer and James M. Zwiener 0899	Effects of natural and induced environments on silver/Inconel reflecting surface coatings for FEP Teflon film.	S0069
LONG DURATION EXPOSURE FACILITY EXPERIMENT M0003-5 THERMAL CONTROL MATERIALS Charles J. Hurley p961	Physical and optical performance of gold, silver, and aluminum mirrors and metallized films.	M0003-5
ION BEAM TEXTURED AND COATED SURFACES EXPERIMENT (IBEX) Michael J. Mirtich, Sharon K. Rutledge, Nicholas Stevens, Raymond Olle and James Merrow p989	Functional durability and properties of ion beam textured silicon, titanium, copper, Inconel, and stainless steel materials exposed to the space environment.	
ELLIPSOMETRIC STUDY OF OXIDE FILMS FORMED ON LDEF METAL SAMPLES W. Franzen, J. S. Brodkin, L. C. Sengupta and P. L. Sagalyn p1005	Optical constants of six metals (aluminum, copper, nickel, tantalum, tungsten and zirconium) exposed to space.	M0002-2
PRELIMINARY RESULTS FROM THE LDEF/UTIAS COMPOSITE MATERIALS EXPERIMENT R. C. Tennyson, G. E. Mabson, W. D. Morison and J. Kleiman p1057	Time to outgas, dimensional changes, coefficients of thermal expansion, atomic oxygen erosion and damage due to micrometeoroid/debris impacts on a stainless steel tube.	A0180
SURVEY OF RESULTS FROM THE BOEING MODULES ON THE M0003 EXPERIMENT ON LDEF	Optical properties of anodized and polished aluminum discs.	M0003
H. G. Pippin, Owen Mulkey, Juris Verzemnieks, Emmett Miller, Sylvester Hill and Harry Dursch		
EFFECTS OF SPACE ENVIRONMENT ON COMPOSITE MATERIALS AND THERMAL COATING (A0138-9) Michel Parcelier and Jean Pierre Assié p1163	A sputter-deposited coating consisting of 1000 Angstroms of nickel overcoated with 600 Angstroms of silicon dioxide protected an epoxy/graphite composite panel from atomic oxygen erosion on LDEF.	A0138-9
PATTERNS OF DISCOLORATION AND OXIDATION BY DIRECT AND SCATTERED FLUXES ON LDEF, INCLUDING OXYGEN ON SILICON A. R. Frederickson, R. C. Filz, F. J. Rich and P.	Discoloration pattern on polished single crystal silicon.	M0002-1
EFFECTS OF ULTRA-VACUUM AND SPACE ENVIRONMENT OF CONTACT OHMIC RESISTANCE LDEF EXPERIMENT A0138-11 Jean-Pierre Assié and Alfred Perotto p1607	Comparison of aluminum conductors and contacts with conventional conductors and contacts under different conditions of production and storage in space vacuum. Nickel plated aluminum, silvered copper, and tinned copper conductors.	A0138-11

LDEF Materials Workshop '91 Reference	Metals Related Information	Experiment
LDEF ATOMIC OXYGEN FLUENCE UPDATE Roger J. Bourassa and J. R. Gillis p59	Solar absorptance and thermal emittance of copper grounding straps as function of atomic oxygen and ultraviolet radiation exposure.	
THERMAL CONTROL SURFACES ON THE MSFC LDEF EXPERIMENTS Donald R. Wilkes, Ann Whitaker, James M. Zwiener, Roger C. Linton, David Shular, Palmer Peters, and John Gregory p187	Optical, thermal, physical, and chemical effects of space exposure on silverized Teflon and chromic acid anodized thermal control surfaces.	A0034 A0114 A0171 S0069 S1005
ANODIZED ALUMINUM ON LDEF: A CURRENT STATUS OF MEASUREMENTS ON CHROMIC ACID ANODIZED ALUMINUM Johnny L Golden p211	Solar absorptance and thermal emittance of chromic acid anodized aluminum at various locations on LDEF including pre-flight, and post-flight measurements.	
CHARACTERIZATION OF SELECTED LDEF- EXPOSED POLYMER FILMS AND RESINS Philip R. Young and Wayne S. Slemp p357	A sputter-deposited coating consisting of 1000 Angstroms of nickel overcoated with 600 Angstroms of silicon dioxide protected an epoxy/graphite composite panel from atomic oxygen erosion on LDEF.	A0138-9
LONG DURATION EXPOSURE FACILITY M0003 5 RECENT RESULTS ON POLYMERIC FILMS Charles J. Hurley and Michele Jones p417	Physical and optical performance of gold, silver, and aluminum mirrors and metallized films.	M0003-5
SELECTED RESULTS FOR METALS FROM LDEF EXPERIMENT A0171 Ann F. Whitaker p467	Atomic oxygen effects on multicrystalline silver, cold rolled silver ribbon, copper, molybdenum, and titanium (75A) metal surfaces	A0171
SOME RESULTS OF THE OXIDATION INVESTIGATION OF COPPER AND SILVER SAMPLES FLOWN ON LDEF	Oxidation of silver film on FEP Tetlon through holes in thermal control blankets caused by micro-meteoroid impacts.	
A. de Rooij p479	Thickness of copper oxide layer on grounding straps.	
	Analysis also indicates that silicone contamination of copper straps occurred early in the LDEF mission.	
CHANGES IN OXIDATION STATE OF CHROMIUM DURING LDEF EXPOSURE Johnny L. Golden p49	Oxidation of black chromium plate.	A0076

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# Table 3. Metals Related Information (Continued)

Table 3.	Metals	Related	Information	(Concluded)

NEE	Second	Post-Retrieval	Symnosium

LDEF Second Post-Retrieval Symposium Reference	Metais Related Information	Experiment
LDEF MATERIALS OVERVIEW Bland A. Stein p741	Summary of LDEF findings.	
SPECTRAL INFRARED HEMISPHERICAL REFLECTANCE MEASUREMENTS FOR LDEF TRAY CLAMPS B. K. Cromwell, Capt. S. D. Shepherd, C. W. Pender, and B. E. Wood p1001	Infrared hemispherical reflectance of tray clamp surfaces show essentially no dependence on atomic oxygen fluence. There did appear to be a slight dependence on solar radiation exposure.	
SURFACE CHARACTERIZATION OF SELECTED LDEF TRAY CLAMPS T. F. Cromer, H. L. Grammer, J. P. Wightman, P. R. Young, and W. S. Slemp p1015	Documents changes in the surface chemistry of tray clamps taken from different locations on LDEF.	
SECOND LDEF POST-RETRIEVAL SYMPOSIUM INTERIM RESULTS OF EXPERIMENT A0034 Roger C. Linton and Rachel R. Kamenetzky p1151	Space environmental effects on thin film mirrors including mirrors with silver, osmium, gold surfaces and mirrors with silicon monoxide and magnesium fluoride overcoated aluminum surfaces.	A0034
THE INTERACTION OF ATOMIC OXYGEN WITH COPPER: AN XPS, AES, XRD, OPTICAL TRANSMISSION AND STYLUS PROFILOMETER STUDY Ganesh N. Raikar, John C. Gregory, Ligia C. Christl, and Palmer N. Peters p1169	Analysis of data on a thin film copper sample exposed on Row 9 show that 55 nm of copper was converted stoichiometrically to cuprous oxide.	
LDEF MATERIALS DATA ANALYSIS: REPRESENTATIVE EXAMPLES Gary Pippin and Russ Crutcher p1187	Solar absorptance and thermal emittance of copper grounding straps as function of atomic oxygen and ultraviolet radiation exposure.	

# Other Reports With Metals Related Information

Other Reports With Metals Related Information Reference	Metals Related Information	Experiment
CHANGES IN CHEMICAL AND OPTICAL PROPERTIES OF THIN FILM METALS MIRRORS ON LDEF P. N. Peters, J. C. Gregory and G.N. Raikar	Reports composition and thickness of oxides formed on thin films of Au, Ga, Cu, Ni, Os, Sn, Mo, Al, Ir, and Ge deposited on silica optical flats.	A0114
ANALYSIS OF SYSTEMS HARDWARE FLOWN ON LDEF - RESULTS OF THE SYSTEMS SPECIAL INVESTIGATION GROUP H. W. Dursch, W. S. Spear, E. A. Miller, G. L. Bohnhoff-Hlavacek, and J. Edelman	Exposure on LDEF caused no discernible effect on the bulk microstructure of aluminum alloy 6061-T6.	Tray E10
ATOMIC OXYGEN EFFECTS MEASUREMENTS FOR SHUTTLE MISSION STS-4 Journal of Geophysical Research, Vol. 10, July 1983, pp 569-571	Atomic oxygen reactions on Ag, C, and Os.	
ATOMIC OXYGEN EFFECTS MEASUREMENTS FOR SHUTTLE MISSIONS STS-8 AND 41G NASA Technical Memorandum 1000459 Volumes I, II, III	Protection of composites by thin atomic oxygen resistant metallic coatings (Volume I, p5-1) Atomic oxygen effects on metals (Volume I, p2-2).	

Note: Additional reports from the LDEF Materials Results Spacecraft Applications Conference will be added when available.



Figure 1. NASA on-orbit photgraph showing four unanodized aluminum clamps at the corners of experiment Tray C9.



Figure 2. NASA KSC photograph of unanodized aluminum clamp C09-3 and adjacent CAA treated clamp C08-5 (with paint dot).



Figure 3. Position of Clamp Bolts



Figure 4. Clamp Locations For Comparison Of Bare And Anodized Surfaces



Figure 5. Auger Electron Spectroscopy Profile (Clamp C03-5)



Figure 6. Auger Electron Spectroscopy Profile (Clamp C09-7)



Figure 7. Auger Electron Spectroscopy Profile (Ground Control Clamp No. 4)



Figure 8. Auger Electron Spectroscopy Profile (Clamp C03-6)



Figure 9. Auger Electron Spectroscopy Profile (Clamp C09-2)



Figure 10. Silicon Concentrations On Bolt Head Surfaces Determined by ESCA



Figure 11. Surface Silicon to Iron Ratio Concentration by ESCA as a Function of Location



ANGLE FROM RAM, degrees

Figure 12. Atomic Oxygen Fluence and Carbon Concentrations on Bolt Heads Determined by ESCA



Figure 13. Bolt Locations for AES Profile Study of Silicon Contamination



Figure 14. Average Silicon to Iron Concentration for Control Bolts



Figure 15. Silicon to Iron Concentrations for Flight Bolts Compared with Control Bolts



Figure 16. Silicon to Iron Concentrations on Control and Non-Atomic-Oxygen Exposed Flight Bolts