EXPLORATORY EXPERIMENTAL STUDY ON NEUTRAL CHARGE LOW ENERGY PARTICLE IRRADIATION OF SELECTED THERMAL CONTROL COATINGS

by

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FOREWORD

This program was conducted at the Martin Marietta Corporation facility in Denver, Colorado in the time interval from May to December 1968. Mr. Elmer Streed, Chief, Vehicle Systems Design Branch at Ames Research Center, National Aeronautics and Space Administration, was Technical Monitor. Mr. Dennis Farnsworth was Program Manager and Principal Investigator. Messrs. Malcolm Lillywhite, Paul Pecce, Kenneth Karki, James Cooley and Neal Zaun contributed to the program.

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INTRODUCTION

Laboratory tests of thermal control coatings, as shown by Carroll (1968, Ref. 1) have failed to accurately project actual spacecraft performance of those coatings as reported by flight data outside the Earth's magnetosphere. One condition of the actual space environment that may be responsible for the high rate of coating instability in flight is that the interplanetary solar wind is electrically neutral, that is, it is composed of an equal number of positive and negative particles. Most laboratory tests have utilized positive (proton) beams only. The objective of this program was to study the influence of the neutral beam in laboratory testing. The program was exploratory in nature.

SCOPE

The scope of this program was to perform four exposure tests using two materials with half of the test using neutral particulate beams and half using positive particulate beams. Additionally, the experimental setup was to accommodate the isolation of synergistic effects. The experiment was designed to evaluate the influence of a neutral (protons and electrons) particulate radiation flux on the specimen as opposed to a positive (proton) flux within set radiation levels.

TECHNICAL BACKGROUND

The charge accumulation on an insulating thermal control coating is a function of properties of the incident beam and the surface chemistry of the coating itself. The coating, by its very nature, must be electrically neutral, and of course, in time will become neutral. The problem is seen to be that at discrete moments of time the coating may appear to the environment to be electrically biased. Additionally, charge reactions on the surface and

within the material compete with the net effective charge at the surface/beam interface.

If the insulating coating is exposed to a particulate radiation beam consisting solely of positive particles (protons), the surface will tend to charge to a potential as positive as the energy of the beam and thus repel the incident beam. The emission of secondary electrons from the surface also contributes to increase the net positive charge. Within the material, volume ionization and electron-ion pair production lower positive charge build-up. In laboratory tests, small specimen substrates are normally attached to a sample holder at ground potential which may limit the positive charge build-up (in this case the specimen is an oscillator and alternately is being charged and discharged). The net potential attained under these positive beam conditions may therefore, during a test, be somewhat less than the incident beam, but would continually attempt to approach the potential of the incident beam. The concern is that under these conditions the specimen under test may not fully allow the exposed radiation to actually impinge on the surface under test.

Consequently, most investigators have utilized grounding devices to accommodate impingement of the exposure radiation on the surface under test. This may be adequate to control charge build-up. It is nevertheless a different condition on the surface of the coating in relation to the conditions of actual space flight.

Exposing the surface in the laboratory to a beam of particulate radiation consisting of an equal population of protons and electrons (an electrically neutral beam) should more adquately simulate flight conditions. Secondary electron emission would still tend to affect a net positive charge but the conditions in time at the surface should approach real environments.

Obviously, the geometry and chemistry of the coating surface, as well as flux, energy, etc. of the incident beam, all affect the rate and degree of charge build-up. The resultant charge build-up was not only suspected to control ultimate damage caused, but may even modify the damage mechanism. A quantitative evaluation of all these parameters contributing to the charge effect would necessarily entail quite an extensive testing program. Alternately, an exploratory program could be conducted to evaluate the relative magnitude of the charge problem. In the planning stages of this program, the exploratory approach was selected.

A number of neutralizing experiments had been conducted by other investigators prior to the effort attempted here. Meckel and Harkins (1960, Ref. 2) made and neutralized a large diameter particulate beam. Work to neutralize beams in space ion rocket propulsion performed by Pearlstein, Rosenbluth and Stuart (1962, Ref. 3), Michelsen and Kaufman (1963, Ref. 4) and Hubach (1964, Ref. 5) concluded that engine beams could be successfully neutralized. The NASA SERT I flight experiment, reported by Gold, Rulis, Maruna, and Hawersaat (1964, Ref. 6) and Nieberding and Lovell (1966, Ref. 7) successfully confirmed that beam neutralization occurred. Recently, Meckel and Lebduska (1967, Ref. 8), (1968, Ref. 9) constructed apparatus for neutralizing beams for various research applications. Fogdall, Cannaday, and Brown (1968, Ref. 10) experimented with neutralizing a twenty KeV proton beam in a combined environments thermal control coating program. These programs and studies directly contributed to the technology necessary for the program reported here; however, two major problems confronted the study team for this program: neutralization of a solar wind type proton beam adquate for thermal control exposures had not been previously attempted and techniques to identify that the beam was actually neutral had not been identified.

EXPERIMENTAL SETUP

The exposure test for this program was conducted in the Martin Marietta Combined Environment Facility. This facility comprises a vacuum system, a particle accelerator with beam diagnostic equipment, an ultraviolet system, a spectral radiometer, a vacuum integrating sphere, an ambient integrating sphere, a spectrophotometer and a data logger. A description by Farnsworth (1968, Ref. 11) of the standard operational procedure for the complete facility is contained in a Martin Marietta Report No. 1610-68-48. Figure 1 is a general view of the facility.

The central vacuum system includes the exposure chamber, particle accelerator, mass separator, mass spectrometer and the vacuum integrating sphere. Pumping is accomplished with a 500 liter/second vac-ion pump. Rough pumping was accomplished with sorption pumps, however, initial pumping was by a LN_2 trapped mechanical pump.

The particle accelerator utilizes an RF source to generate hydrogen ions. The ionized hydrogen beam passes into a Bennett tube (RF type mass separator) in which the heavier ions are removed to provide a pure proton beam. Following mass separation the protons were accelerated to test energy, and thermal electrons, as required, were injected into the accelerated beam. A mechanical flipper shielded two specimen positions from the particulate radiation.

The electromagnetic source was a high pressure xenon short arc lamp. The electromagnetic energy entered the chamber through two separate 1-inch diameter Suprasil quartz windows so that two of the four specimen positions were exposed to ultraviolet radiation.

Four individual sample holders (Figure 2) were located in the main chamber. Separate cooling and temperature monitoring was provided at each specimen location.

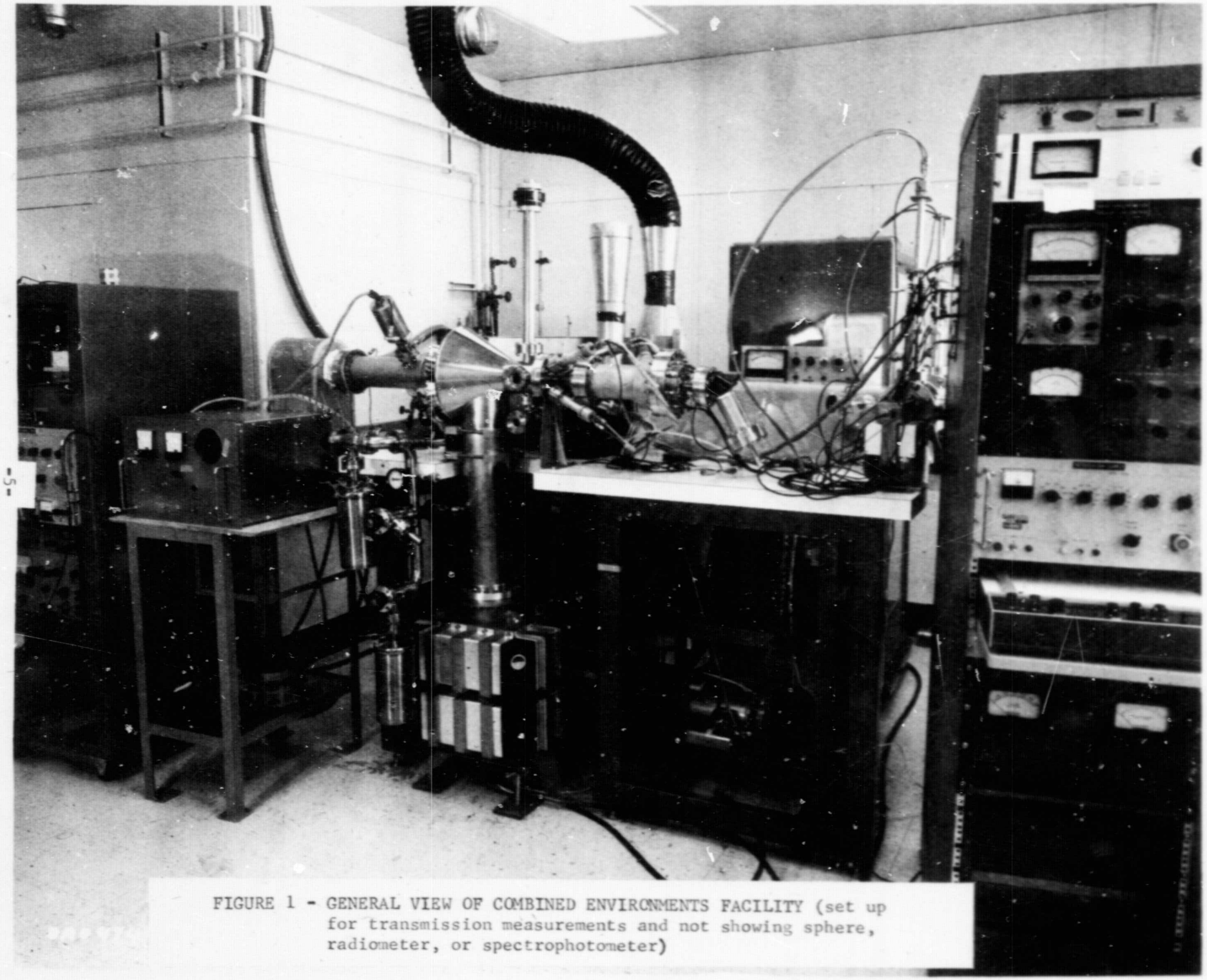

FIGURE 1 - GENERAL VIEW OF COMBINED ENVIRONMENTS FACILITY (set up for transmission measurements and not showing sphere, radiometer, or spectrophotometer)

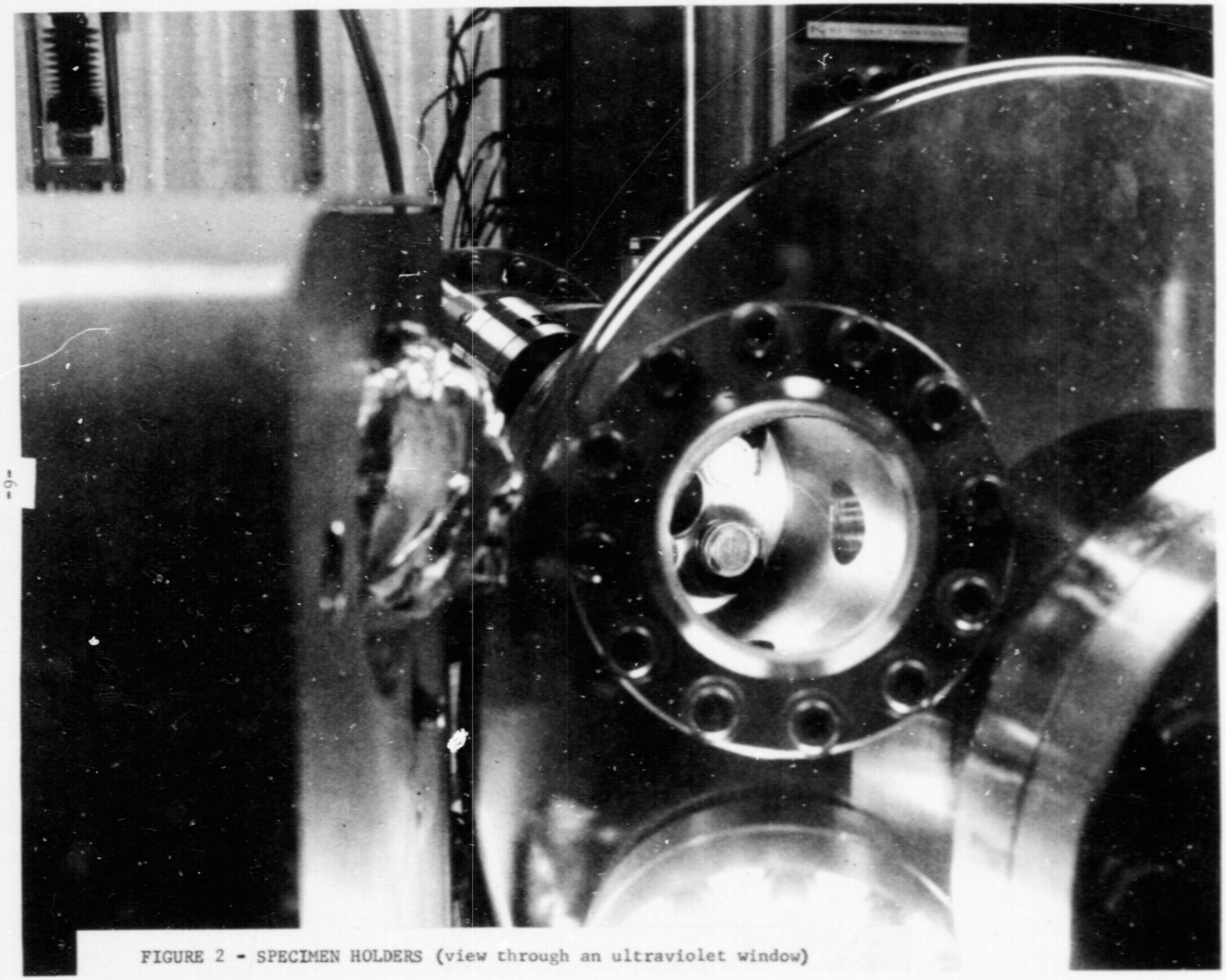

FIGURE 2 - SPECIMEN HOLDERS (view through an ultraviolet window)

A specimen transfer assembly transfers the specimens individually to the vacuum integrating sphere.

The vacuum, center mounting integrating sphere was coupled to a Beckman DK-2A spectrophotometer. The sphere is valved off from the main chamber and maintained at vacuum by a separate ion pump when the specimen chamber is open, so that the integrity of the smoked MgO sphere coating is maintained. A separate integrating sphere was used for making measurements at atmospheric conditions.

EXPERIMENTAL CONDITIONS AND OBSERVATIONS

The four specimen locations accommodated the following conditions:

Position A	No radiation exposure.
Position B	Electromagnetic radiation exposure.
Position C	Particulate radiation exposure (protons alone or protons plus electrons).
Position D	Combined electromagnetic and particulate radiation exposure (with protons alone or protons plus electrons).

Vacuum and temperature conditions are identified as being the same for all four specimen positions (but obviously the surface of specimens exposed to electromagnetic radiation is understandably higher, approximately 2°C, in temperature).

The test environmental conditions are summarized in Table 1.

Vacuum in the specimen chamber is determined from current readings on the main and integrating sphere vac-ion pumps. The pressure is, of course, higher during exposure due to hydrogen gas being bled into the ionization tube. It is suspected that all the hydrogen was not ionized during this study, since possible internal leak areas existed at the ionization tube and mass separator

TABLE 1 - ENVIRONMENTAL CONDITIONS

Exposure Test Number	Test #1				Test #2				Test #3				Test #4			
Pigment	ZnO				ZnO				Al_2O_3				Al_2O_3			
Specimen Position	A	B	C	D	A	B	C	D	A	B	C	D	A	B	C	D
Radiation Environment		UV	H+ e^- UV	H+ e^- UV		UV	H+	H+ UV		UV	H+ e^-	H+ e^- UV		UV	H+	H+ UV
Vacuum during Measurement	1×10^{-8} Torr															
Vacuum during Exposure	8×10^{-5} Torr															
Specimen Temperature Based on Substrate Measurement and Substrate Control	$294^oK \pm 5^o$ except for test #1 position B & D, where higher temperatures are suspected based on specimen appearance after completion of the test.															
Flux Density ($x10^{10} p/cm^2/sec$)		2.4	1.2			2.4	1.2			2.3	1.1			4.0	.9	
Fluence ($x10^{15} p/cm^2$)		4.0	2.0			4.0	2.0			6.1	2.9			11.0	2.2	
Energy	1 KeV															
Approximate Neutralization, %		55	100							30	100					
Proton Specie	H+															
Irradiation Level																
Total Sun Irradiance/Hour	6															
UV Sun Irradiance/Hour	4															
Irradiance																
Total Sun Hour Equivalents	450															
Total Ultraviolet Sun Hour Equivalent	300															

Specimen Description

Material	Source	Comments
ZnO	New Jersey Zinc Co., SP-500 99.9% pure; 0.25-0.35 μ particle	P.B.R. - 5.2 Ball Milled with K_2SiO_3 4 hrs., sprayed 6 coats, overnight dry @ 20^oC, oven cured 1 hr @ 150^oC 6 mil thick
Al_2O_3 (α)	Linde Division - Union Carbide Co. - 99.98% pure, 1.0 μ particle	P.B.R. - 2.0 Ball Milled with K_2SiO_3 2 hrs., (same cure as above), 5.0 mil thick
K_2SiO_3	Sylvania Electronic Products (35% solids) PS-7	

interface within the vacuum system. Furthermore, it has not been established that all hydrogen would be ionized in even an ideal system. The excess hydrogen gas may be considered a contaminant in the specimen chamber.

Specimen temperature is based on substrate temperature control and measurement. A water filled and cooled bellows is pushed against the back of the substrate on the opposite side of the exposed coating. Temperature measurement is by a thermistor in the face of the bellows assembly. Except for Test 1, specimens B and D, the specimen temperature control is adjudged as adequate, but on these specimens, small cracks were noticed after the exposure was completed, suggesting overheating. The specimen holders were modified prior to the remaining exposures to accommodate better contact between the substrate and specimen heat (bellows cooler) sink.

Vacuum pressure, xenon intensity, and specimen substrate temperature were continuously recorded on the data logger during the test.

Flux density, fluence, and percent neutralization were determined with the Faraday cup. The cup utilized is approximately a one inch long cylinder with a ¼ inch inside cup diameter. The entire cup moves across the horizontal diameter of the specimen exposure chamber. The test specimens are positioned approximately one inch to the lee side of the Faraday cup. In scanning across the beam during an exposure, the Faraday cup passed in front of the specimens. Several different modes of operation are available with the Faraday cup, which involve the biasing or grounding of the grid at the entrance of the cylinder, the walls of the cylinder, and the plate that forms the bottom of the cylinder.

Measurements to determine proton flux density were made with plate and grid grounded and the cylinder biased 67.5 volts negatively. The measurements should be accurate since secondary electrons, due to incident protons, could not leave the plate (being driven back by the negative cylinder) and also

when thermal electrons were in the beam, they could not get past the negative cylinder to the plate. The possibility of protons being drawn to the cylinder and away from the plate by the negative bias was examined by varying the bias from -22.5 to -90 volts. No change in proton current readings occurred under these conditions indicating that divergence of the proton beam was not occurring.

A non-uniform flux density existed in the specimen chamber and across the test specimens. The measurement utilized for control and data purposes is that assigned to the center of the test specimens, where reflectance also was determined.

Measurements to determine neutrality were made by biasing the Faraday cup plate 22.5 volts positive and grounding the cylinder walls and grid. Measurements under these conditions may be somewhat in error since secondary electrons arising in the system, Faraday cup grid, and cylinder may also be counted. Table 1 shows differing flux density (and therefore different fluence) between specimens exposed in the same test. This condition resulted from non-uniformity in the particulate radiation source, making the flux density and fluence less on one side (the combined environments side) of the exposure chamber in relation to the opposite side.

The thermal electron source controls are separate from the proton controls, so that 100% neutralization on the combined environment side, as measured by a zero net current reading, would result in a deficiency of electrons on the opposite side (particulate radiation only) by virtue of a greater proton density (more protons than electrons) at the particles only location. Thus, in tests where electrons were utilized, the combined environment side was neutralized 100% by setting the electronics so that an equal number of protons and electrons

were available to that side. This in turn left the opposite side with less than equal electrons, i.e., 55 and 30% neutralized.

A Hy-Cal P8400 radiometer (total irradiance), with and without an RG-2 Schott ultraviolet filter, was used to determine total irradiance of the xenon source and transfer optics. Irradiance was measured outside the specimen chamber but relative to sample position. Final appearance of the Al_2O_3 pigmented specimens exposed to the ultraviolet indicates some non-uniformity across the specimen. Mass purity of the beam was evaluated prior to the exposure, using the mass spectrometer to establish the retardation voltage necessary to eliminate mass two and three protons. Comparable retardation voltages were then used thoughout the exposure to produce a pure proton beam. Retardation measurements of the beam also taken before the exposure test, indicated a good confidence level in the beam energy as reported in Table 1.

As a final observation on experimental procedures, it is worth emphasizing that the techniques used here employed a large diameter beam for particulate radiation as opposed to the approach of pulsed method of scanning a small beam over the specimens under investigation. In addition to the non-scanning approach, the beam is evaluated as being constant since ions are extracted from the plasma by means of a DC electrostatic field and the RF modulation of beam amplitude has been measured (with the Faraday cup) to be less than five percent of the total beam current.

Also, the thermal electron source necessary to inject electronics into the proton beam had to be located away from the electrostatic field utilized in the proton apparatus, (and in fact, required shielding) and the electrons required injection into the beam by means of a negatively biased screen behind the tungsten filament.

DATA ANALYSIS

The data obtained through exposure to the different environments is presented in terms of change in reflectance at a given wavelength. The patterns observed are sufficiently different for the ZnO pigment and the Al_2O_3 pigment so that each material constitutes a separate entity.

The raw reflectance curves for non-neutral exposures compare favorably to data obtained by Cooley (1967, Ref. 12), Streed (1968, Ref. 13), and as documented by Johnson and Boebel (1968, Ref. 14), indicating that the specimens and general testing technique are state-of-the-art. This, of course, does not suggest accuracy but rather consistency.

The data presented in Figures 1 through 6 are in terms of spectral reflectance changes. The abscissa data baseline is the pre-exposure measurement and $\Delta\rho$ is the increase or decrease caused by the exposure as indicated by the post-exposure measurement. Ambient condition and in situ measurements are presented separately.

Figures 3 and 4 are the separate environmental influences on the zinc oxide in potassium silicate coating with reflectance measured at ambient conditions. Figures 5 and 6 are the corresponding influences as measured in situ. The recovery effects of the air measurement in zinc oxide pigments have been previously documented by MacMillian, Sklensky, and McKellar (1966, Ref. 15) and is confirmed by this data.

The in situ reflectance changes occurring with the zinc oxide pigmented coating vary considerably with wavelength. An increase in reflectance below the band gap is of interest. Protons alone, not surprisingly, produced coloration at all wavelengths except below the band gap. The addition of electrons to the proton beam increased coloration at the band gap but also bleached the visible and near infrared. The same general tendency is observable in the

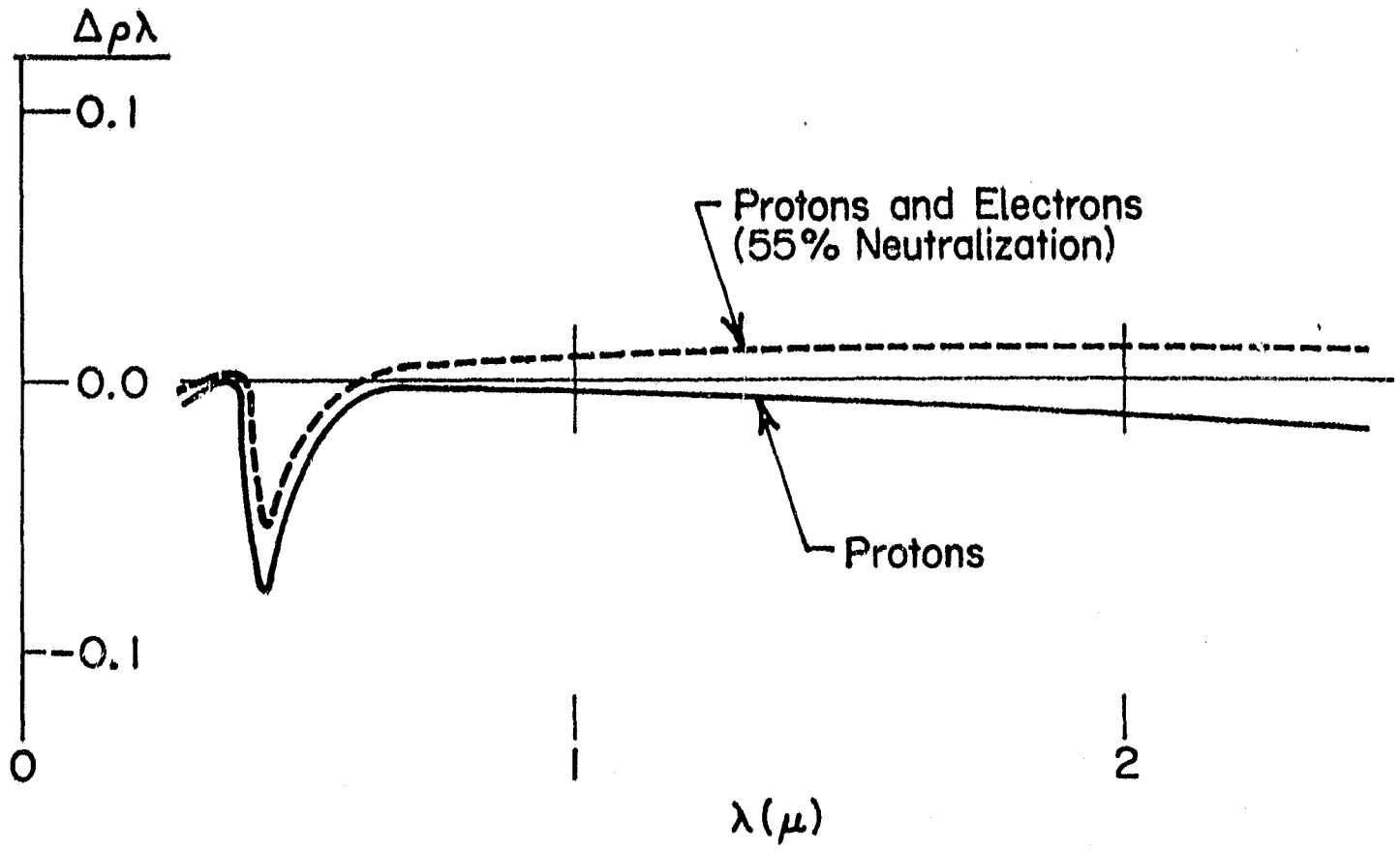

FIGURE 3 - CHANGE IN SPECTRAL REFLECTANCE OF ZnO IN K_2SiO_3, MEASURED IN AIR

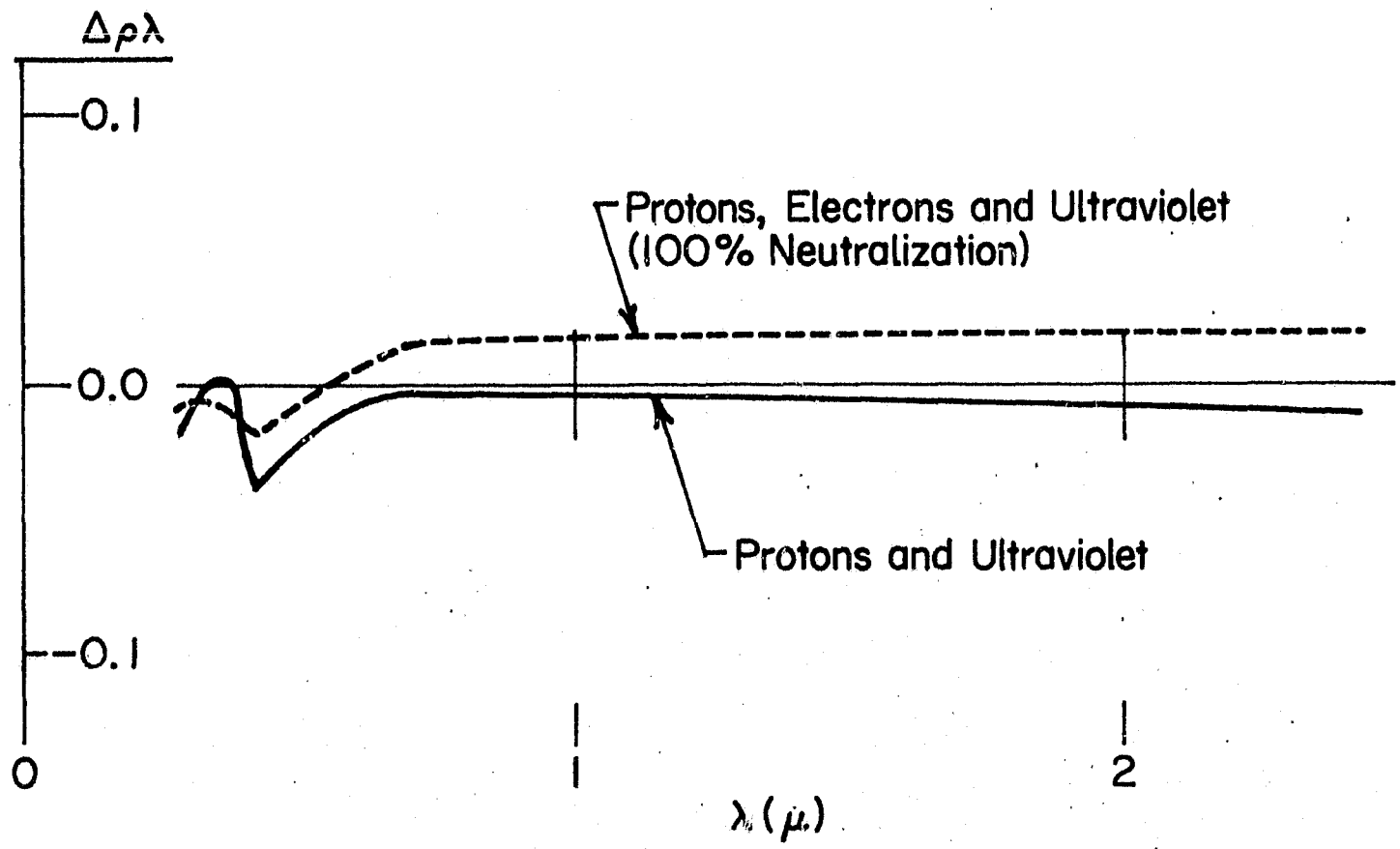

FIGURE 4 - CHANGE IN SPECTRAL REFLECTANCE OF ZnO IN K_2SiO_3, MEASURED IN AIR

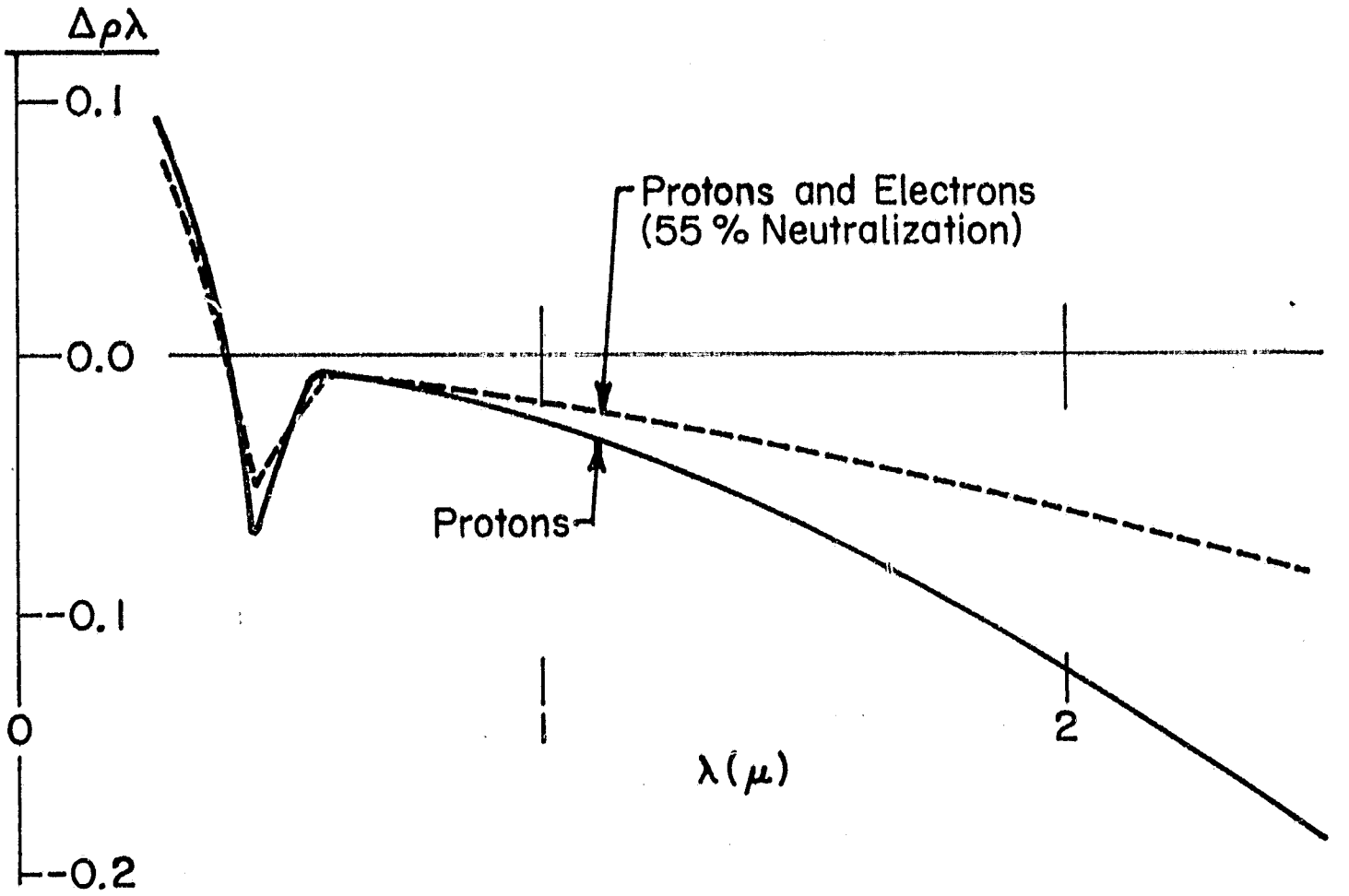

FIGURE 5 - CHANGE IN SPECTRAL REFLECTANCE OF ZnO IN K_2SiO_3, MEASURED IN SITU

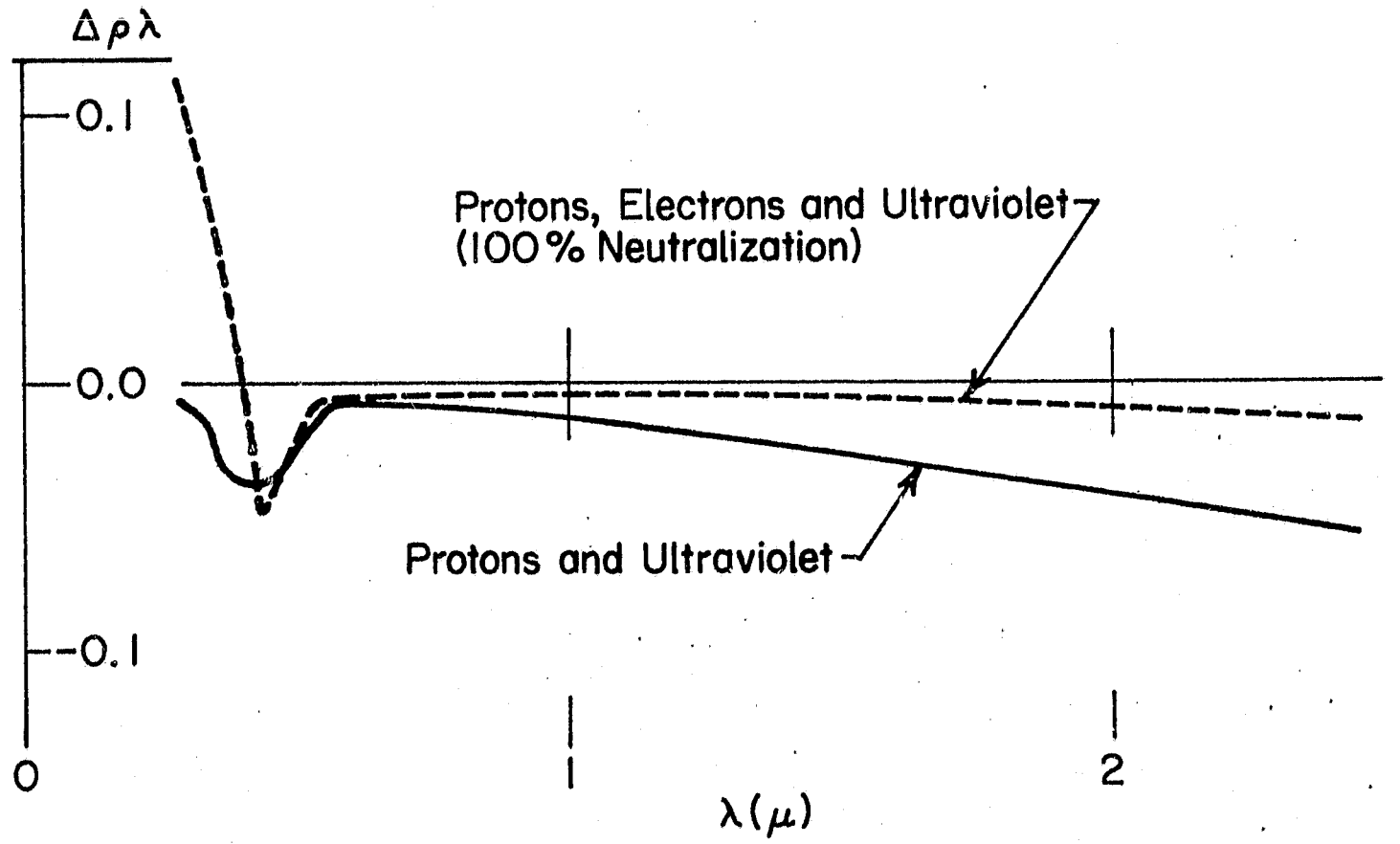

FIGURE 6 - CHANGE IN SPECTRAL REFLECTANCE OF ZnO IN K_2SiO_3, MEASURED IN SITU

combined environment exposures, however, it should be remembered that specimen overheating was suspected in the test where electrons were added to the combined environment test. In all the zinc oxide specimens, the addition of electrons is seen to cause less change in reflectance than when the particulate radiation was all protons. In the post test air measurements, recovery (bleaching) is seen to occur in the visible and infrared regions, the pattern below the band gap is reversed, and the reflectance damage at the band gap appears permanent.

There is no apparent significance in the small differences between the ambient (Figure 7) and in situ (Figure 8) measurements in aluminum oxide. The differences observable are most likely due to systematic or random errors, and are adjudged to be not of influencing magnitude.

A predominant reflectance change occurs between 0.30 and 0.40 microns. Protons and ultraviolet have the effect of coloring this region and electrons have the effect of bleaching this region. As with the zinc oxide, the pattern is that the addition of electrons enhances the stability of reflectance.

The 0.40 micron region in aluminum oxide is not at the band gap. The reflectance change has the characteristics of a color center in that the magnitude of change is an apparent function of radiation. It could be either a "physics" color center, i.e., belonging to the general F or V center classification, or a "chemical" color center, i.e., a function of the appearance of a new chemical impurity form as a result of ionization, oxidation, or migration of an original impurity in the material. Of related interest is that Levy (1961, Ref. 16) has reported observing an absorption band in this same region, introduced by gamma rays, while working with "pure" Al_2O_3 crystals.

Tables 2 and 3 summarize the integrated solar absorptance values. The tables show the magnitude of change in integrated solar absorptance values

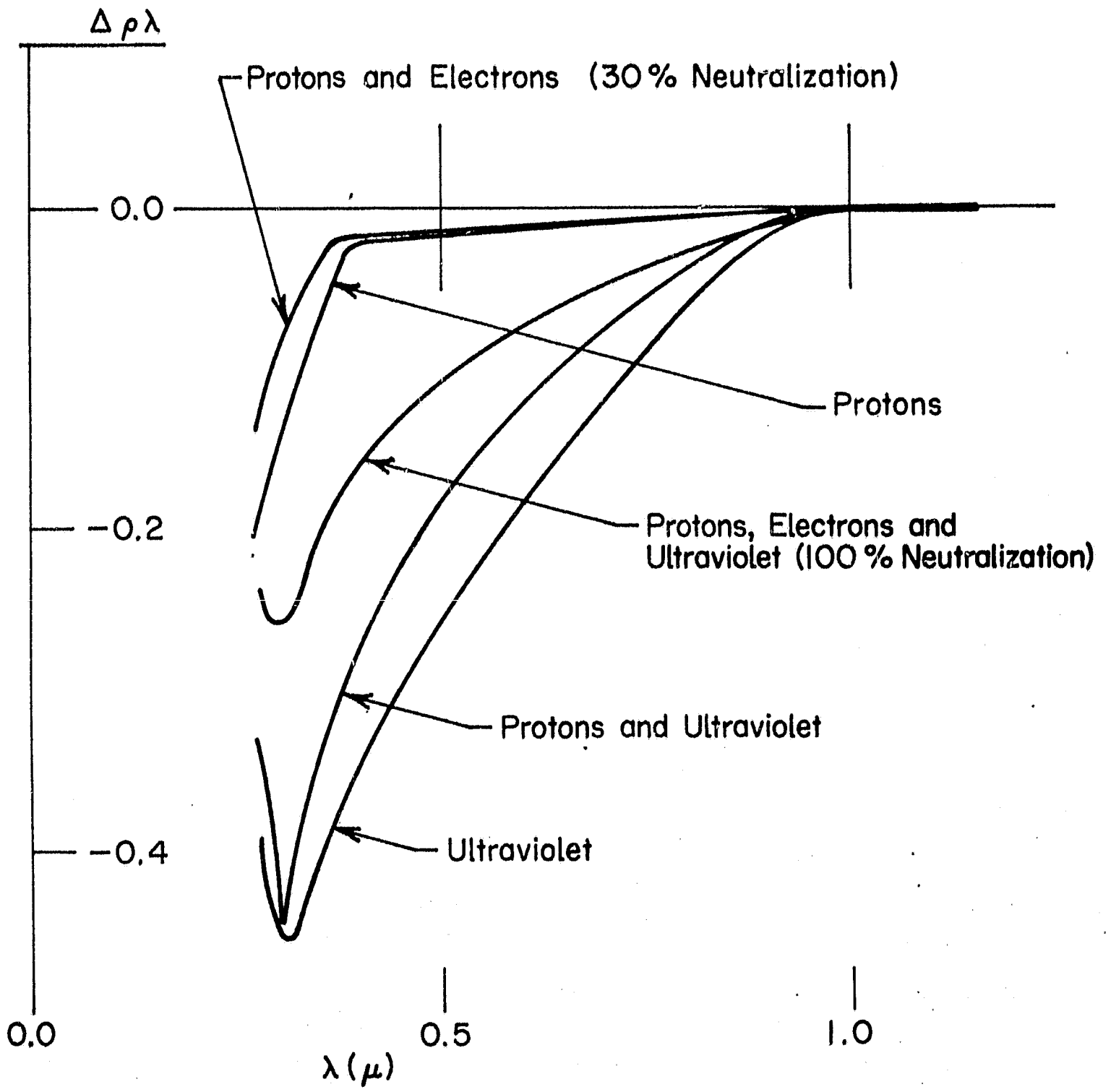

FIGURE 7 - CHANGE IN SPECTRAL REFLECTANCE OF Al_2O_3 IN K_2SiO_3, MEASURED IN AIR

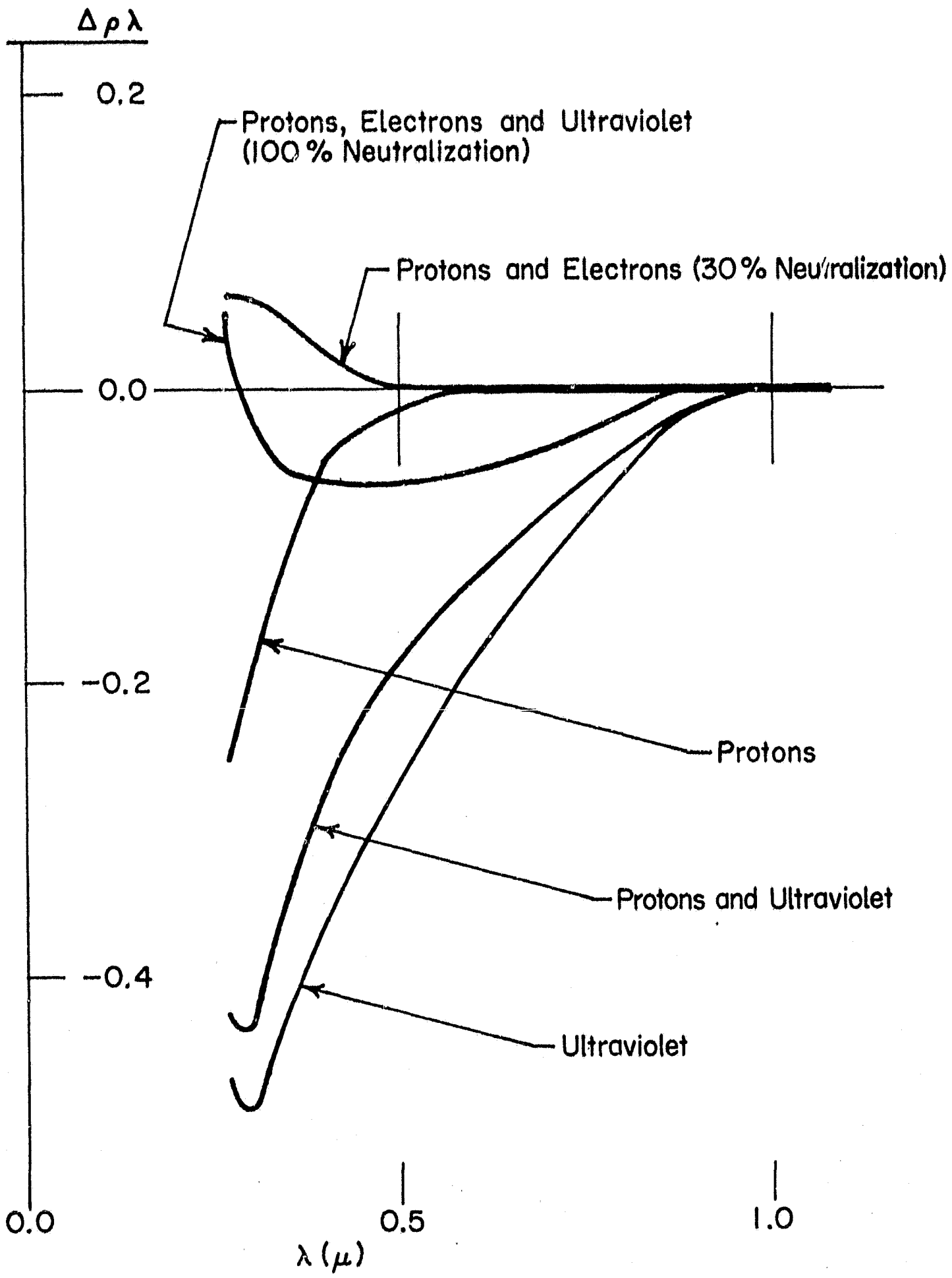

FIGURE 8 - CHANGE IN SPECTRAL REFLECTANCE OF Al_2O_3 IN K_2SiO_3, MEASURED IN SITU

TABLE 2 - UNEXPOSED SOLAR ABSORPTANCE VALUES

	$\bar{X}$ Pretest in Air	Range	$\bar{X}$ Pretest in Situ	Range
ZnO	.18	.17 - .19	.18	.18
Al_2O_3	.08	.07 - .08	.10	.08 - .11

TABLE III - INCREASE IN FRACTIONAL SOLAR ABSORPTANCE VALUES AS A RESULT OF ENVIRONMENTAL EXPOSURE

Environmental Condition	ZnO $\Delta\alpha_s$ in air	ZnO $\Delta\alpha_s$ in situ	Al_2O_3 $\Delta\alpha_s$ in air	Al_2O_3 $\Delta\alpha_s$ in situ
Ultraviolet	0.01	0.02	0.15	0.15
Protons	0.02	0.03	0.03	0.02
Prontons and Electrons	0.00	0.03	0.02	0.00
Protons and Ultraviolet	0.01	0.02	0.11	0.10
Protons, Electrons, and Ultraviolet	0.00	0.02	0.06	0.04

normally of interest to design people. The same pattern, as described above, exists in the spectral data; the values are comparable with other investigators', the expected differences between the air and in situ differences exist, and the apparent beneficial tendency of adding electrons to the exposure test is seen.

CONCLUSIONS

This program was exploratory in nature. The conclusions suggested are therefore tentative and should be sustained by subsequent studies. Also, no relationships of the effect of electrons to other radiation or testing conditions are postulated. The suggested conclusions are:

1. No obvious relationship has been identified in this study to explain the disparity reported between flight and laboratory thermal control stability data.
2. Introduction of thermal electrons into the laboratory testing environments is a factor in coating damage as indicated by spectral reflectance changes. The observable influences of thermal electrons under the conditions utilized are:
   a. In a zinc oxide (semiconductor) pigmented potassium silicate coating the major effect is bleaching in the visible and infrared.
   b. In an aluminum oxide (dielectric) pigmented potassium silicate coating the major effect is bleaching of what is probably a color center in the near ultraviolet.

ACKNOWLEDGEMENT

The thermal electron source used in this program was designed by J. Cooley of the Martin Marietta Corporation.

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