ATOMIC OXYGEN DOSIMETRY MEASUREMENTS MADE ON STS-46 BY CONCAP-II

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ABSTRACT

With increasing flight duration and the possibility of a permanent facility in space, long-term monitoring of material degradation due to atomic oxygen is increasing in importance. Reliance on models to determine the fluence of atomic oxygen is not only necessarily complex but also imprecise due to the strong dependence of oxygen concentration on day/night, latitude and solar activity. Mass-spectroscopy, the traditional method for determining the gas phase species densities at low pressure, is not only expensive but is limited in the area that it can monitor. Our group has developed a simple and inexpensive dosimeter to measure the atomic oxygen fluence via the change in resistance as the sensor element is gradually oxidized. The sensors consisted of thin-film circuit elements deposited on a suitable substrate. Four-point resistance measurements were used to monitor the change in resistance.

Results obtained using silver and carbon dosimeters flown on STS-46 (CONCAP II-01) will be discussed.

INTRODUCTION

In the region of Earth's atmosphere where the shuttle (and future space station) orbit, the composition of the atmosphere ceases to be dominated by diatomic molecules and is increasingly populated by atoms and ions. In particular, the number of oxygen atoms exceeds 90% of the species present in this region. As a dominant species and a major determinate of material degradation of external surfaces in space, atomic oxygen is an important long-life factor for spacecraft operating in this region. Therefore, it is essential that the spatial and temporal variations of atomic oxygen be monitored. Due to the strong dependence of atomic oxygen concentration on day/night, latitude and solar activity the reliance on models to determine the fluence of atomic oxygen is not only necessarily complex but also imprecise, due to a lack of experimental data.[1-6] The traditional method for experimentally determining the gas phase species densities at low

pressure is mass-spectroscopy. A flight MS instrument is not only expensive but also limited in the area that it can monitor. There is a need for a simple, relatively cheap atomic oxygen monitor to monitor atomic oxygen over the long-term.

To meet this need, the Material Science Laboratories, in conjunction with the Consortium for Materials Development in Space at the University of Alabama in Huntsville, is conducting a series of flight experiments. The aim is to develop an instrument that measures the atomic oxygen concentration via the change in resistance as the sensor element is oxidized. The principle of operation is based on the effect that atomic oxygen has on exposed materials. For example, silver reacts very readily with atomic oxygen to form silver oxide

$$2Ag + O \text{ -----> } Ag_2O$$

(it has not been conclusively established whether Ag_2O, AgO or a mixture of both is actually formed) which can be taken to be an insulator in comparison to silver. The above reaction has been found to proceed with an efficiency of 1. That is, each atomic oxygen atom impacting with the surface reacts to form an oxide. These properties appear ideal for development of an atomic oxygen sensor relatively free of many of the problems inherent in the measurement of reactive species concentrations by mass spectrometry. In fact, it has proven to be a simple and very specific technique which dates from early sounding rocket launches undertaken in the 1970's. These experiments used silver films[7-10] to obtain measurements of the atomic oxygen concentration to altitudes up to 95 km. However, due to the atomic oxygen density, these flights were capable of obtaining only one or two data points per flight.[8]

The aim of this work is to develop a dosimeter capable of measuring the hyperthermal atomic oxygen flux variations throughout a long-term space mission. This requires that the sensors be sufficiently sensitive and yet remain viable over the length of the mission. As shown later, under these circumstances the very reactivity of silver can become a problem, even at the lower atomic oxygen densities expected at higher orbits. This indicates a need to investigate other possible candidate materials. By virtue of the orbital velocity of the spacecraft, the relative kinetic energy of atmospheric atoms hitting the front surfaces of a spacecraft is about 5eV per atom (~ 500 kJ mole-1). This is similar in magnitude to chemical bond energies and many examples of enhanced reactivity have been reported under these conditions. Effects have been shown to vary from heavy erosion in the case of polymers, carbon and osmium, corrosion of silver and copper, and significant changes in the optical, thermal and electrical properties of most material surfaces.[11-16]

Oxidation rates obtained from two earlier flight experiments, EOIM-2 on STS-8 (1983) and experiment A0114 on LDEF (1984-1990)[11-16] indicated that carbon might provide a simple yet accurate method for measuring the atomic oxygen flux. Carbon, unlike silver, reacts to form a volatile oxide that then dissipates into space. For example,

$$C + O \text{ ----> } CO$$

and the carbon erodes with time, increasing the resistance across the sensor element. Results reported previously[17] indicate that the reactivity of carbon to hyperthermal atomic oxygen is much lower than silver with the reaction probability of atomic oxygen atoms reduced to between 0.1 and 0.15. This effectively increases carbon sensor lifetimes by between 6 to 10 times compared to silver.

To this end, prototype dosimeters were designed using carbon and silver dosimeters, mounted on CONCAP-2 and flown on the Space Shuttle Atlantis (STS-46) in July-August 1992. The results of this experiment are reported here.

EXPERIMENTAL APPROACH

1. Dosimeters

The silver and carbon films were deposited between gold pads on 1 cm square substrates. The films were prepared and supplied by Dr. Renschler (Los Alamos), Dr. Smith/Ms. Lan (McDonnell Douglas) and Metech, PA. The specifications of the dosimeters are given in Table 1. The substrates were mounted in individual gold-coated Airpax integrated circuit mounts with Aramco 569 or 571 high temperature adhesives. Electrical connections within the mounts were made by conventional thermal acoustic bonding methods. Figure 1 shows two carbon and one silver dosimeters (preflight) in their mounts. External leads from the mounts were connected to the CONCAP measuring system.

Table 1
Dosimeter Specifications

Number of Samples	Material	Channel	Thickness	Reactivity
2	Silver	6 & 14	220 nm	1
1	vitreous carbon	24	290 nm	0.1 - 0.15
1	vitreous carbon	5	380 nm	0.1 - 0.15
1	carbon	28	28 μm	unknown

The electrical resistance of the devices was measured during the flight exposure to provide real-time differential flux measurements, using a 4-point contact technique. In practice, however, this turned out to be a 3-point contact system. Sensitivity to contact resistance was therefore not removed.

A dosimeter lasting for multiple orbits moves in and out of the sun repeatedly as it orbits the Earth. This results in periodic changes in the temperature of the sensor elements as the element is alternatively heated and cooled. As both reaction rate and resistance are temperature-dependent, it is essential to characterize the effects of these changes. Platinum resistance thermometers (RTD's) were mounted in the base-plate to monitor changes in the temperature (in addition to controlling the hot-plate used in other experiments).

After final testing of the CONCAP-II controller and measuring system, the samples were mounted at Goddard Space Flight Center. This was done as close as possible to the flight with final changes being made at Kennedy Space Center just before closing the motorized door assembly. Figure 2 shows the preflight experiment insert plate on which the dosimeters are mounted.

2. Flight Description

The exposure was made towards the end of the STS-46 mission, with the shuttle at 123 nautical miles (228 km) altitude. The shuttle was in a nominal 28.5 deg-inclination orbit and, during the CONCAP-2 exposure, was flown at this relatively low altitude to maximize the O-atom exposure during the limited experiment time of 20 hours. The shuttle was oriented so that the normal to the cargo bay was within ±2 deg of the velocity vector. This ensured that all the atmospheric O-atoms

struck the experimental surfaces at close to normal incidence. The gas can was positioned next to the aft bulkhead. Reflection of atoms from this bulkhead may have affected the absolute fluence of O-atoms seen by CONCAP.

Flight operations for CONCAP are given in Table 2. The experimental surfaces on CONCAP-2 were protected from ambient contamination during all ground and orbital operations by a hermetically sealing lid which was operated by the astronauts. The lid was opened as the shuttle maneuvered into the correct attitude and closed before leaving this attitude.

Table 2

Flight Operations	Experiment Elapsed Time
Orbiter in correct attitude Power to CONCAP controller MDA (lid) open at Mission Elapsed Time 5 days:22 hr:42 min	0
Hot plate on	4 hr:3 min
Temporary de-activation of hot plate	4 hr:34 min
Reactivate hot plate	5 hr:33 min
Hot plate off	15 hr:16 min
CONCAP MDA closed, controller off	20 hr:50 min

3. CONCAP-II Hardware

CONCAP-II utilizes a Get Away Special (GAS) carrier system fitted into the Space Shuttle payload bay. The 5-cubic-foot GAS canister has a hermetically sealed motorized door assembly, which protects samples and sensitive surfaces during all ground and flight operations, other than the exposure period itself. CONCAP-II has an electronic experiment controller and a data system to record resistance measurements of up to 32 samples several times per minute during exposure, as well as a hot-plate capable of maintaining samples at 320° C.

CONCAP's power supply and data acquisition/storage system are self-contained, requiring no Shuttle interface, and are adaptable for other kinds of experiments as well. The power system consists of a battery, solid state switches, and DC/DC converters to supply the heater and electronics. The system supplies 1.4 kW-hrs of power at 28 volts. The 80C86 microprocessor-based data and control system is capable of sampling up to 32 analog channels and uses a 12-bit analog to digital converter. Data blocks are stored every 60 seconds in normal mode and every second in high rate mode; time and experiment status are stored in each block as well.

RESULTS

Six dosimeters were flown. They consisted of two silver and four carbon films, (see Fig.1). All the dosimeters worked successfully with the exception of one carbon film that was mounted on the

320°C hot plate. The measured atomic oxygen flux is compared with values calculated using the MSIS-86 model of the atmosphere and preliminary results from the mass spectrometer flown on the same mission.[18]

1. Silver.

Figures 3 and 4 are plots of R_o/R versus elapsed experiment time, where R_o, and R are the initial and real-time resistance's, respectively. These results amply demonstrate the extreme reactivity of silver, the samples being exhausted in less than 30 seconds following the start of data collection (there was a delay of 1 minute after the removal of the cover to prevent false starts). The presence of a passive surface layer of absorbed gas or other contaminants on the surface of a sensor element is known to yield a non-linear initial response.[7] The programmed start-up delay prevented us from defining this response.

Following the criteria established by Henderson and Schiff,[7] results from the two silver samples yielded AO fluxes of 1.7 and 1.64 x 10^{15} atoms/sec. for an atomic oxygen density of approximately 2.15 x 10^{9} atomic oxygen atoms/cm^{-3}. These results are high compared with the average flux calculated for EIOM-3 of 1.4 x 10^{15} atoms/sec (and that predicted by MSIS model). While both films had identical thicknesses of 22 nm, Channel 6 was significantly more oxidized when measurements started. As thermally-accommodated atomic oxygen also readily oxidizes silver, we believe that the apparent high flux can be explained by the presence of thermally accommodated atomic oxygen scattered from the lid and other areas of the cargo bay.

These results indicate that while silver dosimeters are capable of providing a snapshot of the atomic oxygen flux they clearly demonstrate the need to use less reactive materials both for long-term dosimeters and to allow for the differentiation between hyperthermal and thermally-accommodated atomic oxygen.

2. Carbon.

Two forms of carbon were used: thin vitreous carbon films supplied by Dr. Renschler (Sandia) and a commercial thick-film composite supplied by Metech, PA.

Vitreous Carbon.

Results for the vitreous carbon are shown in Figures 5 & 6. The plots of R_o/R extend over four orbits and reveal a more complex picture than for silver. The reversal in the monotonic decrease in R_o/R at periodic intervals is the result of solar heating (and a resulting decrease in resistance). As mentioned earlier, the platinum resistance thermometers (RTD's) mounted in the baseplate were used to measure the change in temperature due to solar heating (in addition to controlling the heaters for the hot-plate). In Figure 7 the effect of solar heating can be seen, modulated by the orbital period. Since the platinum detector elements are inside the 0.25 inch thick aluminum heater plate, this effect (of the order of 10 deg.) may be less, for example, than the solar heating effects on the dosimeter elements. Using the temperature coefficient of resistance we found the temperature rise to be approximately 15 - 20 degrees.

It must be remembered that not only does this change in temperature alter the resistance, it also changes the reaction rate. As a preliminary estimate, a calculation of the temperature dependence of the reaction rate was made using

$$r = Ae^{\frac{-\Delta E}{RT}}$$

and taking the Arrhenius activation energy, ΔE, value of 1200 cal $mole^{-1}$ given in ref.17 and the change in temperature from the RTD's. It was found that the maximum effect of solar heating was insignificant, increasing the rate of reaction less than 3%.

Using the change in resistance, the flux for each film is given in Figures 8 and 9 together with the flux predicted by the MSIS model.[1] These results indicate that after an initial non-linear period, the flux calculated from the change in resistance mirrors the periodic behavior predicted by the MSIS model but that significant differences can be seen with respect to the day/night values obtained. These differences are supported by the preliminary mass spectrometry data (Fig. 10).

It should be noted that although the two films were approximately 90 nm different in thickness both carbon films were completely eroded in approximately the same time. We believe that this can be explained by the closer proximity of the thicker film to the hotplate, increasing the temperature of the sensor and therefore the reaction rate. This conclusion is supported by the apparent increase in flux after the hotplate is switched on, peaking at 1.8×10^{15} and 2.2×10^{15} atoms sec^{-1} for channels 24 and 5 respectively at MET of 340 minutes.

Thick Carbon Film.

A thick (28 ± 5 μm) carbon film supplied by Metech, PA was employed as a possible candidate as a long term dosimeter. Unfortunately, the resistance values fell into a range where the bit noise was significant. Nevertheless, after smoothing, solar effects can still be observed. Figure 11 shows the response of this sensor. The sharp drop in R_0/R, starting at 300 minutes into the mission, results from leakage from the hotplate heating the sample.

From the slope in Figure 11 (before hotplate heating and after the initial non-linear period, 50 to 250 minutes) we calculated the average flux at 3.5×10^{15} atoms sec^{-1}. This result is in very good agreement with both the MSIS and vitreous carbon data.

The hotplate heating was used to obtain a measure of the Arrhenius activation energy, ΔE, of hyperthermal oxygen atoms with this particular form of carbon. This yielded a value of approximately 2600 cal $mole^{-1}$. It must be noted that the temperature during this period never reached a steady-state and thus the activation energy obtained should be taken as an estimate only.

The most notable result from the use of this film is that over the entire mission the resistance changes by only 7-8% for an estimated total fluence of 1.1×10^{20}. This indicates that this sensor is capable of measuring fluences of the order of 1.5×10^{21} atoms.

Summary and Conclusions.

In this paper we report on real-time atomic oxygen flux data obtained on STS-46 using the CMDS autonomous payload system, CONCAP-II. Three different sensors; Ag, vitreous carbon, and a thick-film carbon composite were exposed to the Low-Earth orbit environment. The real-time resistance changes were measured and recorded. The following conclusions were reached:

1) The silver sensors are too reactive, having a very short life under the high flux experienced. A problem also arises because silver is oxidized by both thermally-accommodated & hyperthermal atomic oxygen, making it impossible to distinguish between them.

2) Sensors consisting of vitreous carbon lasted for approximately 4 orbits and a fluence of 2.5×10^{19} atoms. They yielded flux variations that, while generally in good agreement with the MSIS model, indicated significant diurnal differences. A result confirmed by the preliminary mass data. These results indicate the need for further work in refining the computer models.

3) Noisy data and the lack of an Arrhenius activation energy for the thick-film carbon composites limited the conclusions that could be reached in this case. However, the film was found to be very durable, yielding only a 7-8% change in resistance over 20 hrs for a fluence of 1.1 x 10^{20} atoms cm^{-2}.

4) A major point to come out of this study is the importance of temperature effects on resistance based dosimeters. We are still in the process of eliminating temperature effects. The final results will be reported later. However, it is apparent from the data that temperature control is a necessary feature to eliminate changes in resistance and in reaction rates resulting from solar heating or leakage from nearby instrumentation.

5) In the process of developing these sensors the lack of data on reaction rates of materials exposed to 5 eV atomic oxygen became very apparent. More work is needed in this area.

Overall, the results indicate that atomic oxygen dosimeters are capable of playing a significant role in the study and monitoring of the Low-Earth environment replacing in part the traditional method for determining the gas phase species densities at low pressure, mass-spectroscopy - a technique which is not only expensive but limited in the area that it can monitor. To conclude, our group has developed a simple and inexpensive dosimeter to measure the atomic oxygen fluence via the change in resistance as the sensor element is gradually oxidized.

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MSIS data and the preliminary EOIM-MS data was provided by Steve Koontz, JSC.

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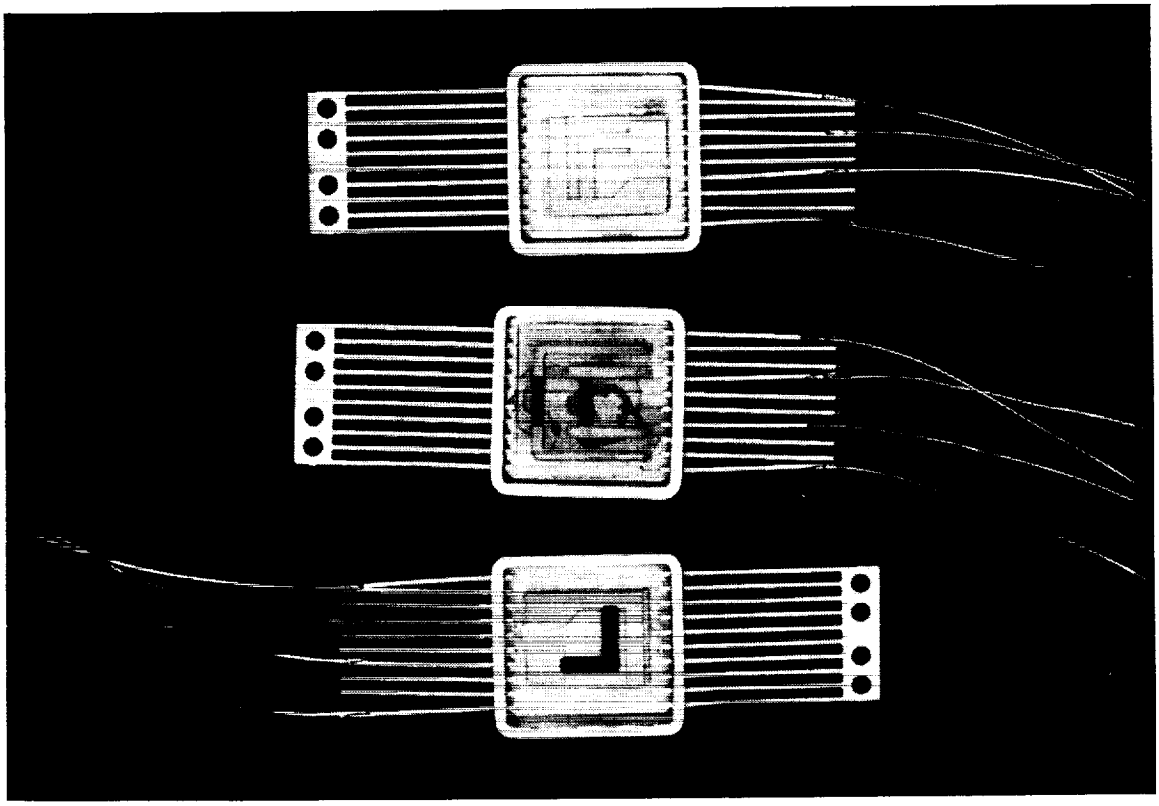

Figure 1: Three pre-flight dosimeters (two vitreous carbon, one silver) in their mounts.

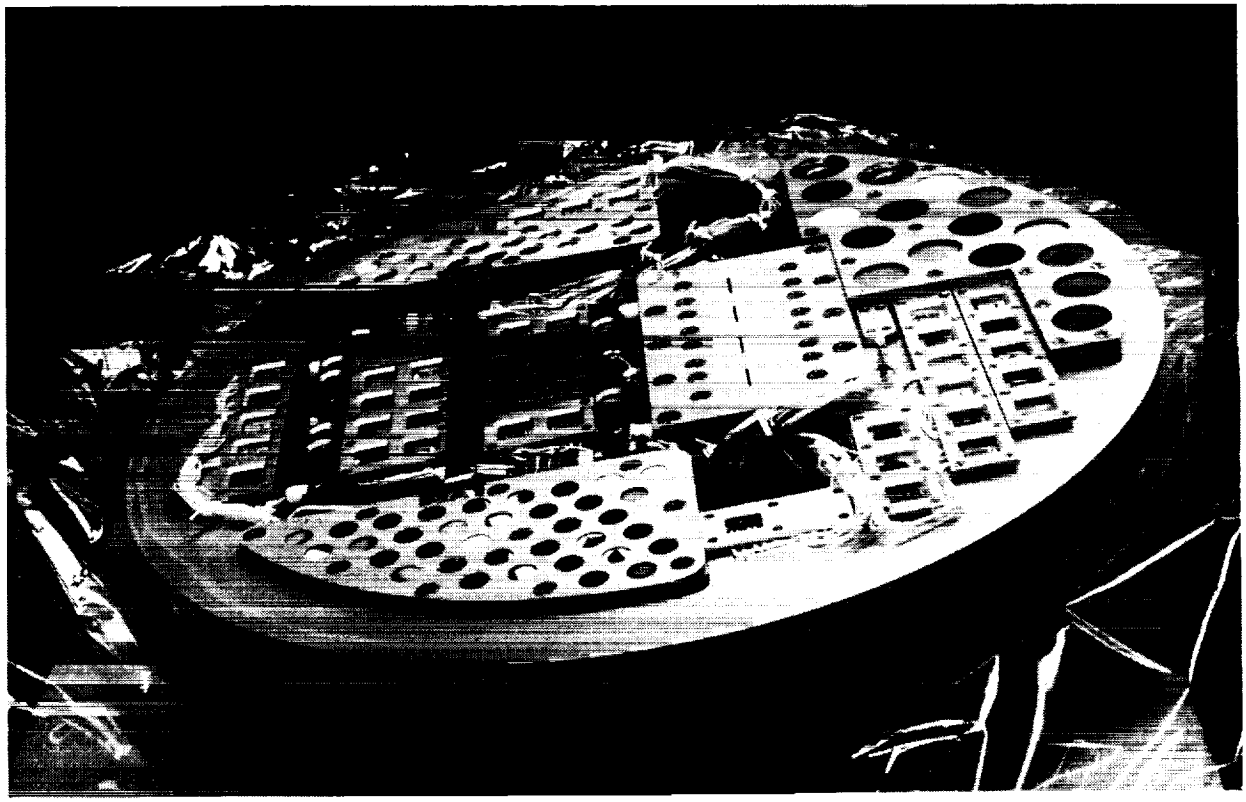

Figure 2: The experimental insertion plate on which the dosimeters were mounted.

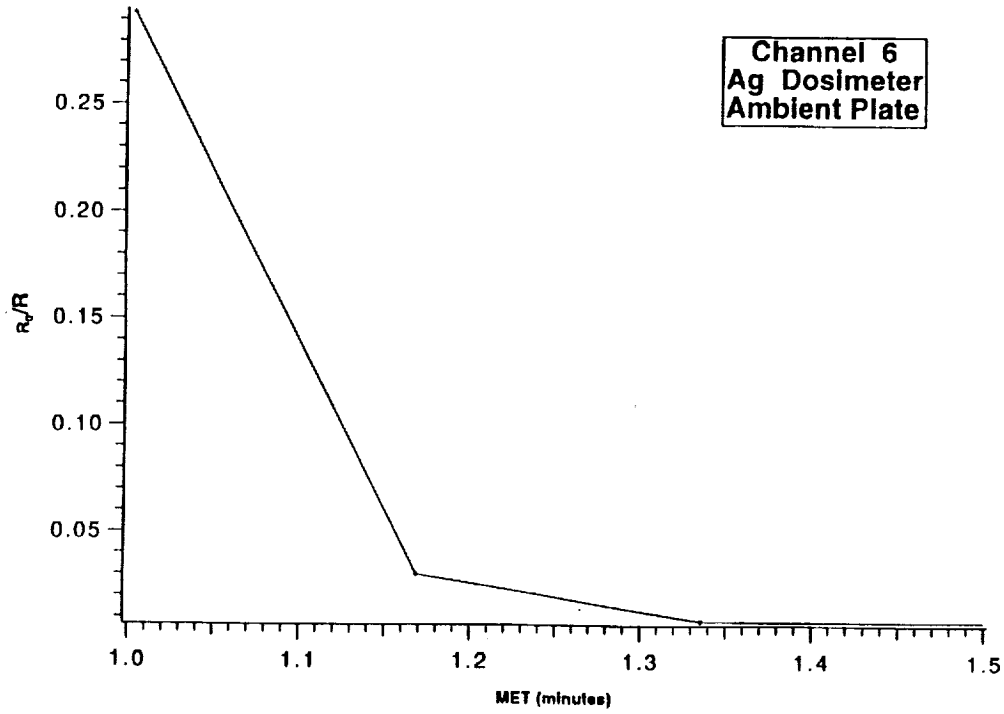

Figure 3: The change in resistance upon exposure to atomic oxygen for Silver dosimeter (Channel 6).

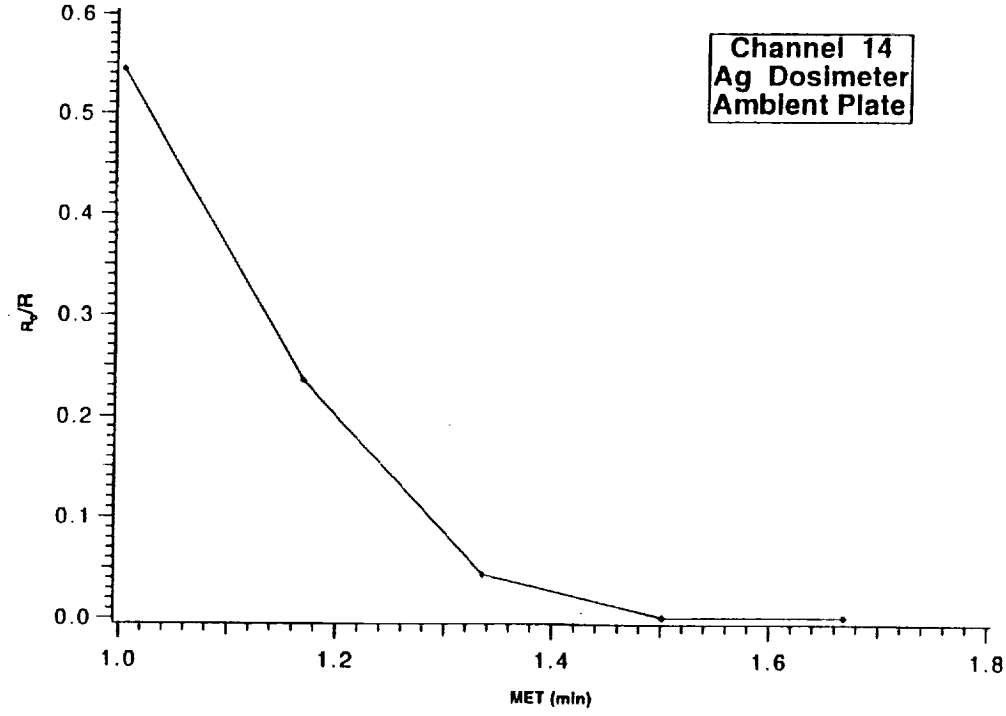

Figure 4: The change in resistance upon exposure to atomic oxygen for Silver dosimeter (Channel 14).

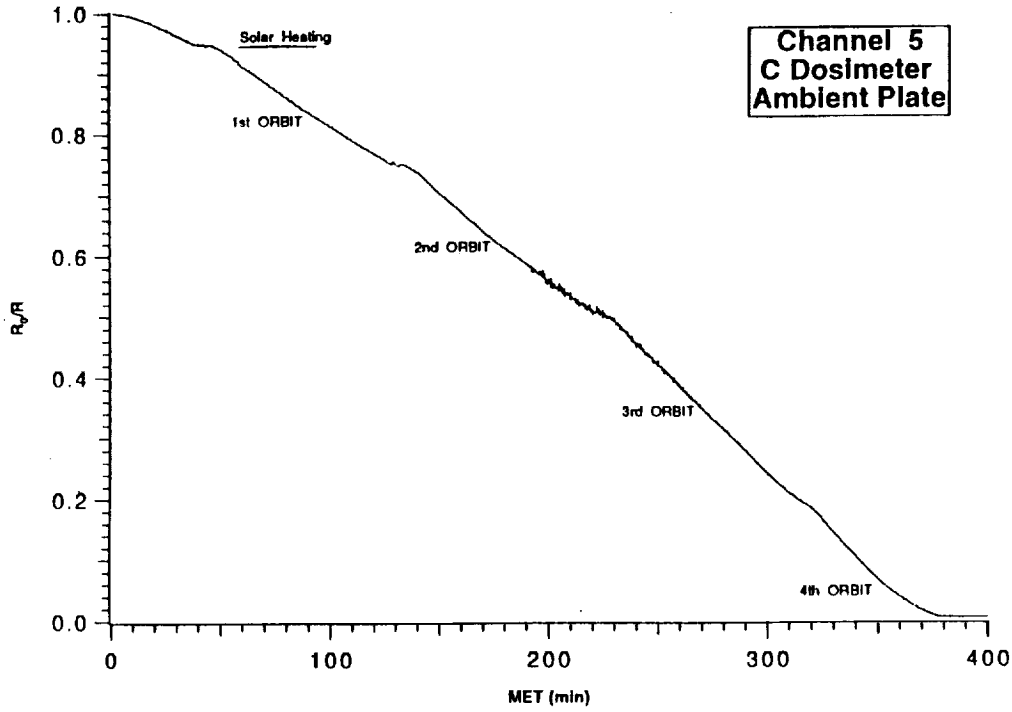

Figure 5: The change in resistance upon exposure to atomic oxygen for the vitreous carbon dosimeter (Channel 5).

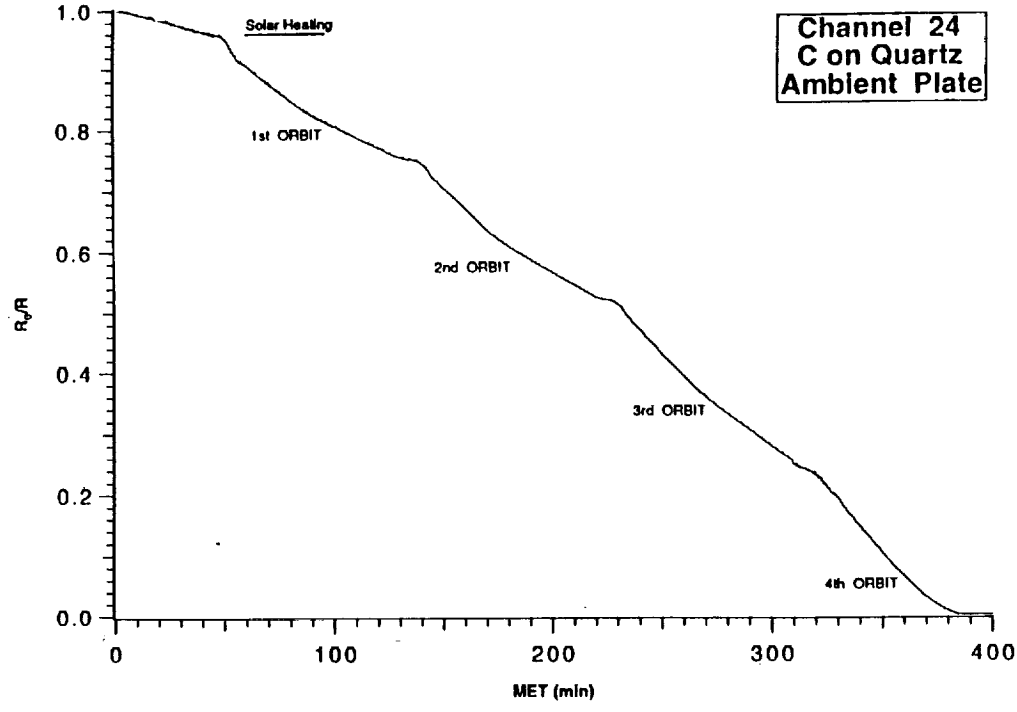

Figure 6: The change in resistance upon exposure to atomic oxygen for the vitreous carbon dosimeter (Channel 24).

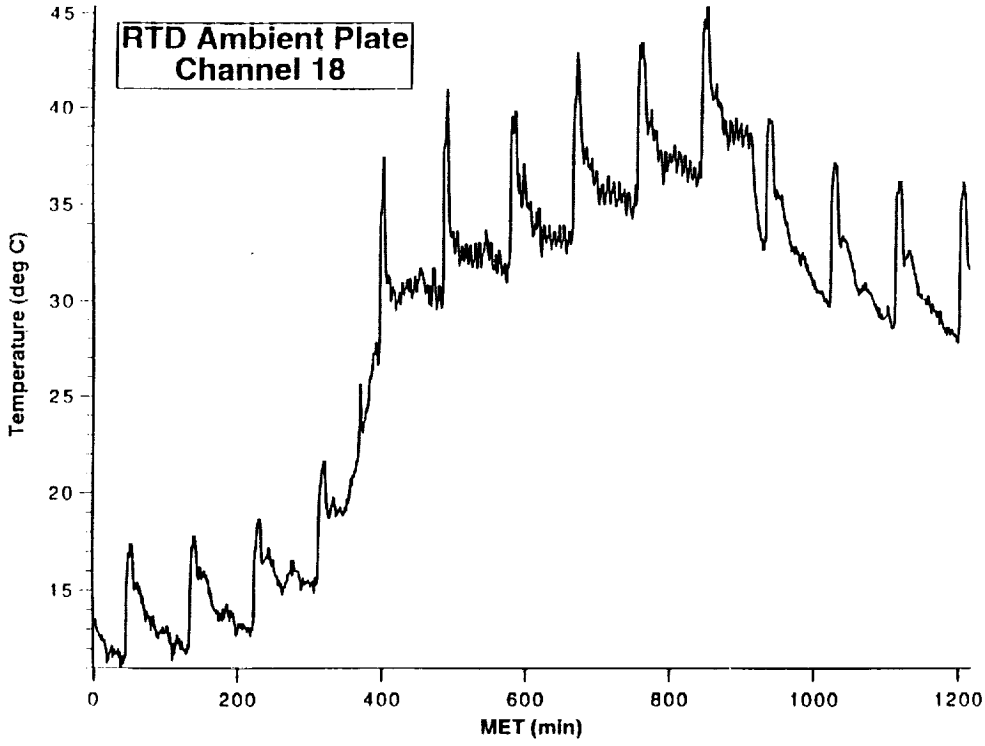

Figure 7: Temperature variations of the baseplate throughout the mission.

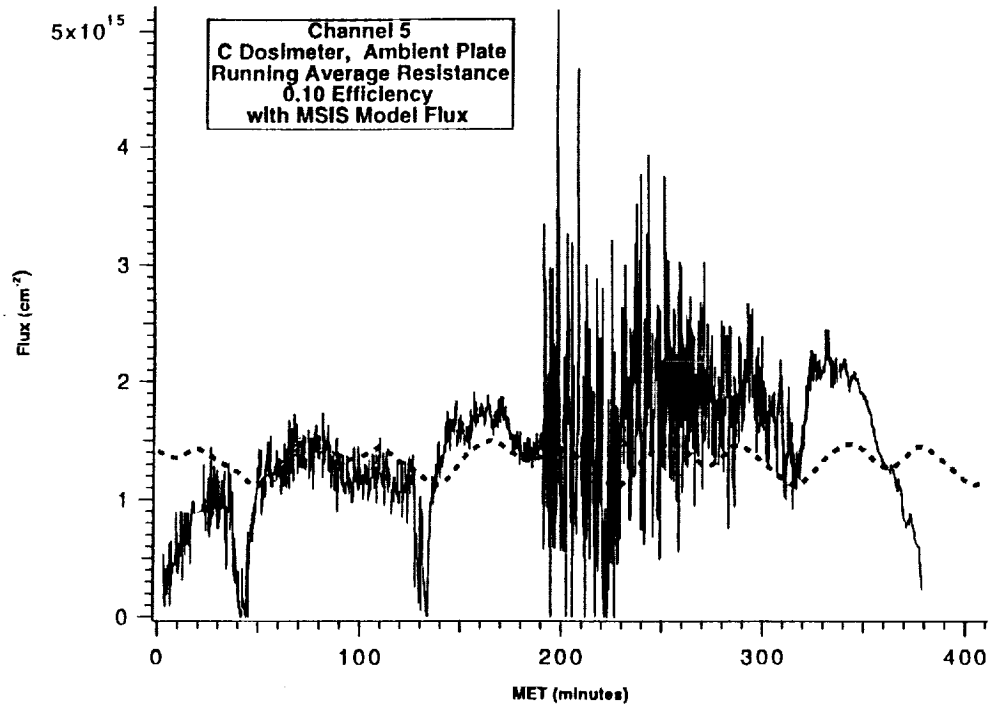

Figure 8: Variation of Atomic Oxygen Flux with time for dosimeter (Channel 5) and MSIS model.

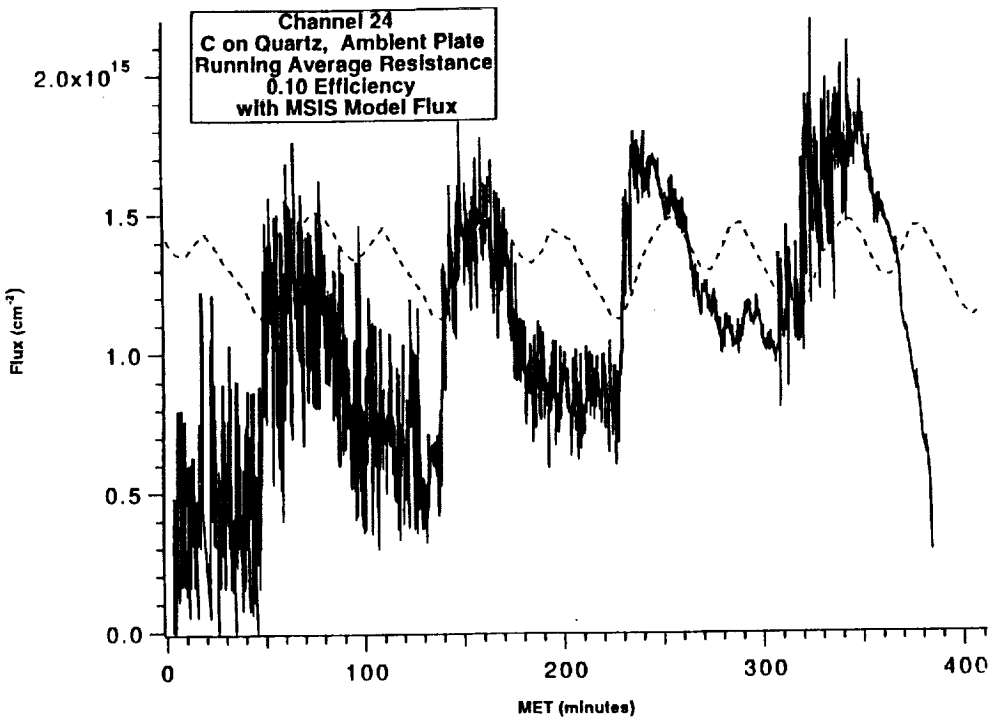

Figure 9: Variation of Atomic Oxygen Flux with time dosimeter (Channel 24) and MSIS model.

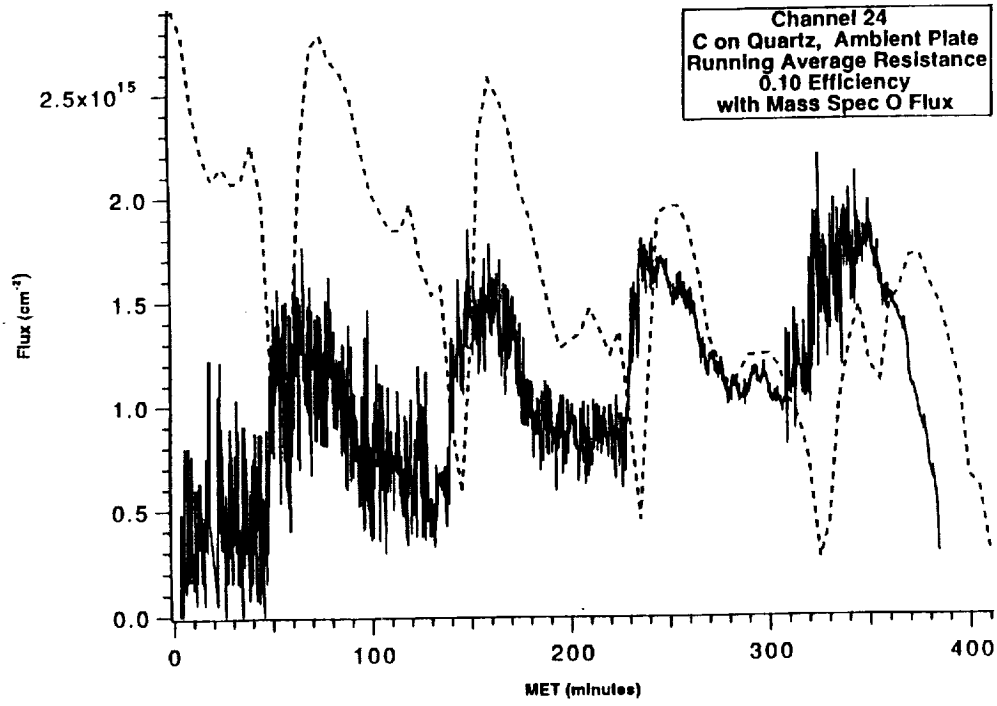

Figure 10: Variation of Atomic Oxygen Flux with time dosimeter (Channel 24) and preliminary mass-spectrometry data.

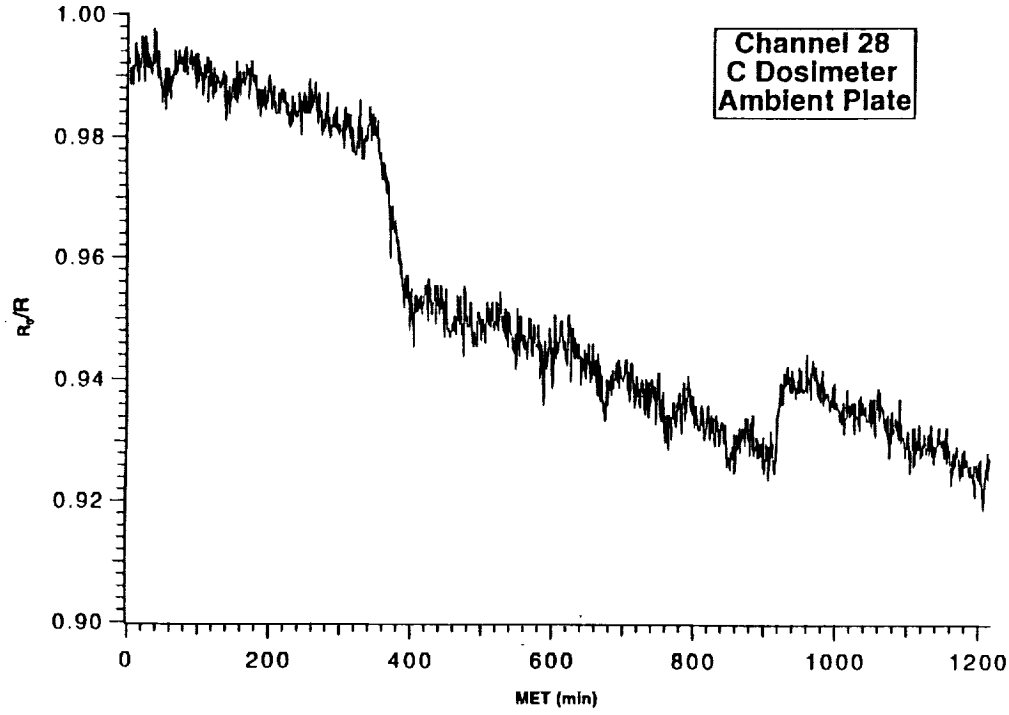

Figure 11: The change in resistance upon exposure to atomic oxygen for the thick-film carbon dosimeter (Channel 28).