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MASS SPECTROMETERS AND ATOMIC OXYGEN

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ABSTRACT

The likely role of atmospheric atomic oxygen in the recession of spacecraft surfaces and in the Shuttle glow has revived interest in the accurate measurement of atomic oxygen densities in the upper atmosphere. The Air Force Geophysics Laboratory is supplying a quadrupole mass spectrometer for a materials interactions flight experiment being planned by the NASA Johnson Space Center. The mass spectrometer will measure the flux of oxygen on test materials and will also identify the products of surface reactions. The instrument will be calibrated at a new facility for producing high energy beams of atomic oxygen at the Los Alamos National Laboratory. This paper summarizes plans for these calibration experiments.

INTRODUCTION

The measurement of atomic oxygen densities in the atmosphere with mass spectrometers is one of the outstanding problems in aeronomy. The measurements were originally made to investigate the structure of the earth's atmosphere. Recently, atomic oxygen has been implicated in the recession of spacecraft surfaces(1-4) and in the Shuttle glow(5,6). These phenomena have renewed interest in the accurate measurement of atomic oxygen densities.

The accuracy of the mass spectrometer measurements is limited by uncertainties in the response of the instruments to the atomic oxygen. The major technical problems are recombination of the atomic oxygen to the molecular species on the inner surfaces of the ion sources, differences in detection sensitivity between thermal and hyperthermal oxygen atoms, energy equilibration of oxygen with ion source surfaces, and background contributions to the mass 16 and 32 signals.

These problems have been addressed in the past by designing the two types of ion sources sketched in Fig. 1. Open ion sources(7) seek to reduce

recombination as much as possible. Much of the atomic oxygen can pass through the electron beam and into the mass analysis section of the instrument without any surface interactions. In contrast, closed ion sources(8) force complete recombination. The electron beam is surrounded by a closed accommodation volume. The atomic oxygen enters this volume through a pinhole. A baffle deflects the oxygen to the walls of the chamber, where it forms molecular oxygen.



The sensitivity of closed source mass spectrometers can be calculated from the measured sensitivity to thermal molecular oxygen and the rate of effusive flow through the pinhole opening(9,10). However, open source instruments must be calibrated to atomic oxygen in the laboratory. Such instruments have been exposed to beams of oxygen atoms formed in microwave discharges(11) or on heated filaments(12). Though high velocities and intensities have been obtained for other gases in mass spectrometer calibration experiments(13), the limitations of the atomic oxygen calibrations have been the low energy and intensity of the beam.

Research groups at the NASA Johnson Space Center (JSC), the Los Alamos National Laboratory, and the Air Force Geophysics Laboratory (AFGL) are engaged in a cooperative space flight experiment designed to measure the effects of atomic oxygen on spacecraft materials. The experiment is called Evaluation of Oxygen Interactions with Materials (EOIM)-III (17). AFGL is supplying a mass spectrometer to monitor the flux of oxygen atoms interacting with the EOIM test materials. This instrument will be calibrated at the atomic oxygen high energy beam facility that has recently been constructed at Los Alamos.

AFGL QUADRUPOLE MASS SPECTROMETER

The AFGL Quadrupole Ion/Neutral Mass Spectrometer (QINMS) is a compact, versatile, fast sampling instrument(15). Its primary purpose is to measure the concentration and the identity of each constituent of the

gas entering its sampling orifice. It is sensitive to neutral and positive ionic species, though not to both simultaneously. The spectrometer design is based on similar instruments flown extensively on sounding rockets. QINMS has been used on one Shuttle flight(16).

In addition to the composition information, the instrument collects a fraction of the total ion current on a grid located between the ion source and the entrance aperture to the quadrupole rods. This current is a measure of the total pressure of the neutral gas in the neutral mode and the total density of the ions in the ion mode. The sampling rates of the composition data and the pressure/density data are both fast enough to resolve such transient effects as ionospheric irregularities and spacecraft thruster engine firings.

The ion source of the AFGL instrument is drawn to scale in Fig. 2. This entire assembly is mounted on top of the quadrupole housing, and is contained inside a metal cylinder. An O-ring sealed cover is retracted in flight to expose the top grid of the ion source to the external environment. This ion source is somewhere between the open and closed extremes described above. The grids crossing the path of the incoming oxygen provide some surfaces for recombination, while still allowing some of the oxygen to reach the quadrupole region unimpeded.



FIG. 2: Scale drawing of the ion source used in the AFGL mass spectrometer. The source incorporates features from both the open and closed designs.

ATOMIC OXYGEN GROUND CALIBRATIONS

The energetic oxygen atom source that has been developed at Los Alamos employs a Continuous Optical Discharge (COD) technique(17,18). A 1.5 kW cw CO_2 laser is focused to a 0.02 cm diameter spot within the throat of a supersonic expansion nozzle. A pulsed CO_2 laser, aligned co-axially with the cw beam, is fired once to start a discharge in the nozzle. The cw laser then pumps the discharge continuously, maintaining temperatures of 15,000 to 30,000 K within the throat of the nozzle. In the source chamber, molecular oxygen is seeded into a noble carrier gas, such as neon or helium to reduce the power required for breakdown. The oxygen is completely dissociated by the discharge and is accelerated to high velocity by collisions with the carrier gas during the expansion.

The COD atomic oxygen beam source is mounted to the Los Alamos Molecular Beam Dynamics Apparatus (LAMBDA) (19). Three stages of differential pumping separate the source from the primary experiment chamber. LAMBDA is equipped with several sophisticated beam diagnostic tools, including a differentially pumped quadrupole mass spectrometer, a time-of-flight (TOF) chopper, and recently, a spinning sphere absolute pressure gauge(20). Recent advances in the development of the atomic oxygen beam facility are discussed in another paper in this session (Cross et al.).

The most difficult aspect of the calibration experiment is measuring the absolute flux of the oxygen beam. Beam diagnostics such as the differentially pumped mass spectrometer would need calibration in the same way as the flight instrument does. Fig. 3 is a rough sketch of the experimental layout that we have chosen for the mass spectrometer calibration experiments. In essence, the idea is the same as the closed ion source concept. The O-atom beam enters the main chamber from the source on the right. On the far side of the main chamber, approximately one meter away, the beam enters a smaller accommodation chamber through a small aperture of known area. The flight mass spectrometer is mounted on the beam axis on the far side of the accommodation chamber.

FIG. 3: Experimental layout of the Los Alamos atomic oxygen high energy beam facility to be used for the mass spectrometer calibration experiments.



Two beam flags, F1 and F2, can be used to block the beam. F1 is between the first and second differential pumping stages in the source, and is used to turn off the beam completely. F2 is located inside the accommodation chamber and is designed to deflect the incoming beam to the walls. With F2 blocking the beam, the spinning sphere absolute pressure gauge will be used to measure the pressure rise in the accommodation chamber when the beam is turned on. We assume that the atomic oxygen recombines completely and thermalizes on the walls of the accommodation chamber (and choose wall materials to ensure this). The measured pressure rise and the known rate of effusive flow from the small aperture are then used to calculate the absolute intensity of the beam entering the chamber.

Finally, when both flags are removed from the beam, the flight mass spectrometer will be exposed to the full flux of atomic oxygen in the molecular beam. A typical calibration experiment will first involve measuring the absolute intensity and composition of the beam using the LAMBDA beam diagnostics and the techniques described above. Comparison of the known beam characteristics to the intensities of the mass 16 and 32 peaks (0 and 0, respectively) will be used to derive the sensitivity of the flight instrument to atomic oxygen along with the degree of recombination. The effects of surface conditioning and length of exposure to oxygen atoms, as well as the influence of beam velocity, beam intensity, and carrier gas concentration will be observed.

In a preliminary experiment, a thermal beam of oxygen molecules (formed in the supersonic expansion with the laser turned off) was directed into the mass spectrometer ion source. Mass spectra taken with the beam flagged and not flagged appear in Fig. 4. The 32 amu peak is the parent oxygen, and 16 amu is atomic oxygen produced by dissociative ionization. No atomic oxygen was present in the beam. The mass 4 peak is the helium carrier gas. Additional peaks in the spectra are due to vacuum chamber contamination and air leakage. These include water (18, 17 and 16 amu), nitrogen (28 amu) and molecular oxygen (32 amu). When the beam was chopped with a 400 Hz tuning fork chopper and phase sensitive detection of the mass spectrometer signals was used, only beam components were detected.

SUMMARY

The Quadrupole Ion/Neutral Mass Spectrometer, designed and built by the Air Force Geophysics Laboratory, is a versatile instrument well-suited to measurements within the Shuttle environment. Its configuration is derived from similar instruments designed for sounding rocket flights, and successfully collected data during the STS-4 flight. We plan to operate the same instrument on the Evaluation of Oxygen Interactions with Materials (EOIM)-III experiment presently under development by the Johnson Space Center. This experiment is the third in a series of Shuttle-based experiments designed to measure the effects of atomic oxygen on materials and surfaces. The mass spectrometer will measure the flux of atomic oxygen incident on the materials samples, and will also look for surface reaction products. The data from the experiment will be invaluable in designing spacecraft, such as Space Station, for long duration excursions into the low Earth orbital environment.

A critical part of preparing the mass spectrometer for the flight is calibrating it with a well-characterized high energy beam of atomic oxygen. This will be done at the Los Alamos National Laboratory, where a new source of high energy oxygen atoms has been developed. Using a Continuous Optical Discharge technique, the Los Alamos source produces a beam of oxygen that reproduces many aspects of the space environment accurately. During the calibration experiments, we will investigate the amount of recombination that occurs within the mass spectrometer ion source, the effect of extended exposure to the oxygen beam, and the effects of the beam energy and intensity on the calibrations.



FIG 4: Unmodulated mass spectrometer signals due to a thermal beam of molecular oxygen in helium carrier gas. With the beam off (F1 in beam), the mass spectrometer only sees chamber background species such as water, nitrogen and oxygen. With the beam on (F1 removed from beam) the 0 and 0 signals increase markedly. The 0 species is produced only from dissociative ionization of the parent molecular oxygen.

REFERENCES

1) Leger, L.J., Spiker, I.K., Kuminecz, J.F., Ballentine, T.J. and Visentine, J.T., "STS Flight 5 LEO Effects Experiment-Background Description and Thin Film Results," AIAA Paper 83-2631-CP, Oct. 1983.

2) Leger, L.J., Visentine, J.T., and Kuminecz, J.F., "Low Earth Orbit Atomic Oxygen Effects on Surfaces," AIAA Paper 84-0548, Jan, 1984.

3) Visentine, J.T., Leger, L.J., Kuminecz, J.F. and Spiker, I.K., "STS-8 Atomic Oxygen Effects Experiment," AIAA Paper 85-0415, Jan. 1985.

4) Leger, L.J., Visentine, J.T., and Schliesing, J.A., "A Consideration of Atomic Oxygen Interactions with Space Station," AIAA Paper 85-0476, Jan. 1985.

5) Mende, S.B., Swenson, G.R., Clifton, K.S., Gause, R., Leger, L.J., and Garriot, O.K., "Space Vehicle Glow Measurements on STS 41-D," <u>J. Spacecraft</u> and Rockets, 23, 189-193 (1986).

6) Green, B.D., "Review of the Vehicle Glow," AIAA Paper 85-6095-CP, Nov. 1985.

7) Nier, A.O., Potter, W.E., Hickman, D.R. and Mauersberger, K., "The Open Source Neutral Mass Spectrometer on Atmosphere Explorer -C, -D, and -E," <u>Radio Sci., 8</u>, 271-276 (1973).

8) Pelz, D.T., Reber, C.A., Hedin, A.E. and Carignan, G.R., "A Neutral Atmosphere Composition Experiment for the Atmosphere Explorer -- C, -D, and - E," <u>Radio Sci.</u>, 8, 277-285 (1973).

9) Horowitz, R. and LaGow, H.E., "Upper Air Pressure and Density Measurements from 90 to 220 Kilometers With the Viking 7 Rocket," <u>J.</u> <u>Geophys. Res., 62</u>, 57-78 (1957).

10) Hedin, A.E., Avery, C.P., and Tschetter, C.D., "An Analysis of Spin Modulation Effects on Data Taken With a Rocket-borne Mass Spectrometer," <u>J.</u> Geophys. Res., 69, 4637-4648 (1964).

11) Narcisi, R.S., Schiff, H.I., Morgan, J.E. and Cohen, H.A., "Calibration of a Flyable Mass Spectrometer For N and O Atom Sensitivity," <u>Space</u> <u>Research III, Proceedings of the Third International Space Science</u> <u>Symposium</u>, Washington, DC, April 30-May 9, 1963, Priester, W., Ed., North-Holland, Amsterdam, 1963, pp. 1156-1167.

12) Lake, L.R., Mauersberger, K., "Investigation of Atomic Oxygen in Mass Spectrometer Ion Sources," <u>Int. J. Mass Spec. and Ion Physics</u>, <u>13</u>, 425-436 (1974); Sjolander, G.W., "Atomic Oxygen-Metal Surface Studies as Applied to Mass Spectrometer Measurements of Upper Planetary Atmospheres," J. Geophys. Res., 81, 3767-3770 (1976). 13) Ballenthin, J.O., and Nier, A.O., "Molecular Beam Facility for Studying Mass Spectrometer Performance," <u>Rev. Sci. Instrum., 52</u>, 1016-1024 (1981).

14) Visentine, J.T. and Leger, L.J., "Material Interactions With the Low Earth Orbital Environment: Accurate Reaction Rate Measurements," AIAA Paper 85-7019, Nov. 1985.

15) Hunton, D.E., Trzcinski, E., Wlodyka, L, Federico, G. and Dorian, J., "Quadrupole Ion/Neutral Mass Spectrometer for Space Shuttle Applications," AFGL Technical Report 86-0084, #ADA172000.

16) Narcisi, R.S., Trzcinski, E., Federico, G., Wlodyka, L., and Delorey, D., "The Gaseous and Plasma Environment Around Space Shuttle," AIAA Paper 83-2659, Oct. 1983.

17) Cross, J.B. and Cremers, D.A., "Atomic Oxygen Surface Interactions--Mechanistic Study Using Ground Based Facilities," AIAA Paper 85-0473, Jan, 1985.

18) Cross, J.B. and Cremers, D.A., "High Kinetic Energy Laser Sustained Neutral Atom Beam Source," <u>Nuclear Inst. and Methods in Physics</u>, <u>B13</u>, 658-662 (1986).

19) Pack, R.T., Valentini, J.J. and Cross, J.B., "Multiproperty Empirical Anisotropic Intermolecular Potentials for $ArSF_6$ and $KrSF_6$," <u>J. Chem. Phys.</u>, <u>77</u>, 5486-5499 (1982); Bomse, D.S., Cross, J.B., and Valentini, J.J., "The Dynamics of Infrared Photodissociation of van der Waals Molecules containing Ethylene: An Experimental Study," J. Chem. Phys., <u>78</u>, 7175-7190 (1983).

20) Kern, K., Lindenau, B., David, R. and Comsa, G., "Absolute Determination of Molecular Beam Intensities," <u>Rev. Sci. Instrum., 56, 52-</u> 57 (1985).