PULSED SOURCE OF ENERGETIC ATOMIC OXYGEN

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ABSTRACT

A pulsed high flux source of nearly monoenergetic atomic oxygen has been designed, built, and successfully demonstrated. Molecular oxygen at several atmospheres pressure is introduced into an evacuated supersonic expansion nozzle through a pulsed molecular beam valve. An 18J pulsed CO_2 TEA laser is focused to intensities $> 10^9$ W/cm^2 in the nozzle throat to generate a laser-induced breakdown. The resulting plasma is heated in excess of 20,000 K by a laser-supported detonation wave, and then rapidly expands and cools. Nozzle geometry confines the expansion to promote rapid electron-ion recombination into atomic oxygen. We have measured average O-atom beam velocities from 5 to 13 km/s at estimated fluxes to 10^{18} atoms per pulse. Preliminary materials testing has produced the same surface oxygen enrichment in polyethylene samples as obtained on the STS-8 mission. Scanning Electron Microscope examinations of irradiated polymer surfaces reveal an erosion morphology similar to that obtained in low earth orbit, with an estimated mass removal rate of $\sim 10^{-24}$ cm^3/atom. The characteristics of the O-atom source and the results of some preliminary materials testing studies will be reviewed.

INTRODUCTION

Satellites in low-earth orbit sweep at velocities of ~ 8 km/s through a rarefied atmosphere which consists primarily of atomic oxygen. Experimental pallets flown on Shuttle missions STS-5 and STS-8 clearly demonstrated a dependence of material degradation and mass loss on the ram direction atomic oxygen exposure.[1-4] These experiments indicate that most hydrocarbons and active metals are highly reactive, whereas material containing silicones, fluorides, oxides and noble metals are moderately inert. For Kapton,[®] an important aerospace polymer, it was observed that about one in ten atomic oxygen interactions lead to mass loss due to chemical reaction.[2,3]

The need for continued material degradation studies has been emphasized in a study by Leger et al.[5] where it was demonstrated that atomic oxygen induced material degradation could have a severe impact on the performance of Space Station. The EOIM-3 material pallet, with sophisticated instrumentation to detect atomic oxygen reaction products and to study reaction mechanisms, is scheduled to be deployed on a future Shuttle mission, and NASA is actively pursuing the development of various hardening techniques to make materials more impervious to the effects of energetic atomic oxygen interactons.

The recent setbacks in launch capability have reemphasized the need for a high flux atomic oxygen source which can be used to study material degradation. Although in-flight experiments provide valuable test data, the large

matrix of materials and test parameters, and uncertain flight scheduling now mandate that most of these studies be performed in a ground test facility.
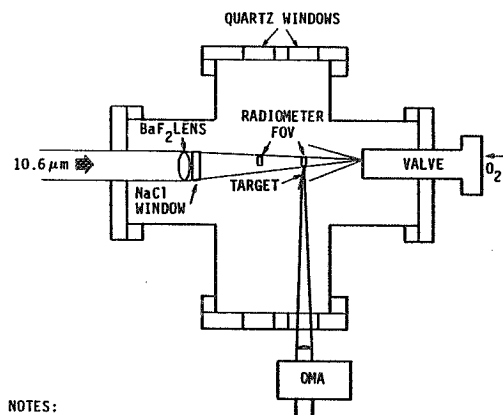
A high flux source of atomic oxygen has been developed at Physical Sciences Inc. (PSI) based on years of research in the area of pulsed laser propulsion.(6-8) The basic concept is to rapidly introduce a burst of gas into an evacuated nozzle and then to focus the output of a pulsed laser to cause a breakdown at the nozzle throat. The subsequent laser-initiated detonation wave will heat the major portion of the gas during the laser pulse creating a high temperature plasma. This plasma will then expand through a nozzle tailored to allow electron-ion recombination but not atomic recombination. As the gas expands its temperature and density will drop, however, its directed velocity increases correspondingly, producing a thermally "cold," high energy beam of oxygen atoms at the nozzle exit. This technique has been utilized to produce a high velocity O-atom source for material degradation studies. For example with 10^{-4}g of gas and a 5J laser pulse we predict formation of $>10^{18}$ oxygen atoms with a characteristic energy of 5 eV. For comparison with other sources, if the source is pulsed at 10 Hz, an average flux of $>10^{17}$ O-atoms/cm^2-s can be maintained on a 100 cm^2 target.

In our research effort at PSI we have constructed a small test facility to demonstrate that high velocity oxygen atoms can be produced. A series of measurements have been performed to demonstrate the presence of atomic oxygen and the measured velocities agreed with theoretical predictions. Measurements of the material degradation of O-atom irradiated targets are being performed, and these experimental observations are described. The design of a larger O-atom test facility presently under construction is also presented.

FACILITY DESCRIPTION

The present test facility consists of a stainless steel high vacuum chamber in which oxygen gas is rapidly pulsed through a conical expansion nozzle and laser heated by a pulsed CO_2 laser to temperatures above 20,000 K.

A schematic diagram of the apparatus is shown in Fig. 1. The vacuum chamber is a standard 20 cm diameter five-way stainless steel high vacuum cross with Con-Flat flanges. Each sidearm extends 20 cm from the center of the chamber. A sixth 10.0 cm diameter port was welded to the bottom of the chamber for evacuation. The top flange of the vacuum chamber has two radiometers for time-of-flight velocity measurements into the chamber. The pulsed valve/nozzle assembly is mounted on the end flange which also contains vacuum feed-throughs for electrical connections. The opposing flange holds the laser focusing lens. Each side

Fig. 1. Schematic diagram of O-atom apparatus

136

flange has two 5 cm diameter quartz view-ports for visual and spectroscopic observations.

The bottom flange is connected to a 10 cm diffusion pump stack equipped with an ionization gauge readout. The ultimate pressure in the chamber is 3×10^{-5} torr. In operation the chamber pressure is kept below 1×10^{-4} torr, to prevent beam interaction with the background gas. (Mean free path at 10^{-4} torr is 50 cm which is greater than the chamber length and is sufficient to provide a "collision free" environment.) The pumping speed of the vacuum chamber is sufficient to allow introduction of a pulse of 10^{-4}g of oxygen at 1 Hz into a background pressure below 1×10^{-4} torr.

The pulsed valve/nozzle assembly is shown schematically in Fig. 2. The valve is a modified Model BV-100V pulsed molecular beam valve from Newport Research, Inc. This valve allows the gener- ation of short duration pulses of gas at high flowrates which cannot be continuously maintained under high vacuum conditions due to pumping speed limitations. The valve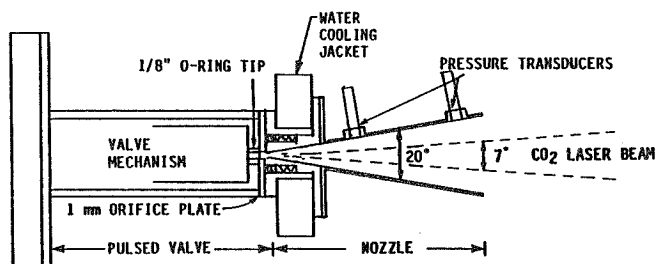 is operated with a 1 mm i.d. orifice plate, and is bolted directly to a 100 mm long, 20 deg full angle aluminum expansion nozzle with a 1 mm i.d. throat. The choked flowrate of the valve/nozzle assembly is 0.19g of oxygen per second per atmosphere stagnation pressure.

Fig. 2. O-atom source pulsed valve/ nozzle assembly

The nozzle has two flush mounted pressure transducers located 43 and 93 mm from the throat with 1 μs response time and 20 mV/psia sensitivity.

A Laser Applications Limited TEA CO_2 laser is used to generate 18J pulses of 10.6μ radiation. The energy is delivered in a 2.5 μs pulse, with approx- imately one-third of the energy delivered in the first 200 ns. The radiation in the gain switched spike generates a laser induced breakdown in the high pressure oxygen at the nozzle throat forming a plasma which continues to absorb the radiation as long as the laser is on.

The laser beam is directed to the test chamber by three gold turning mirrors (Fig. 3). A barium fluoride flat between the second and third mirror reflects eight percent of the beam to a calorimeter to moni- tor the laser energy. A 300 mm focal length barium fluoride lens is used to focus the laser beam to ~1 mm diameter spot size at the nozzle throat.
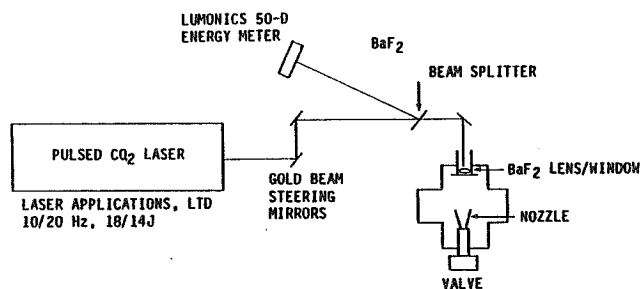

Fig. 3. Laser/optics assembly

BEAM DIAGNOSTICS

Optical measurements have been conducted to characterize the laser initiated O-atom beam. Beam velocities from 5 to 13 km/s were obtained by varying both the stagnation pressure and the laser energy. The velocities were deduced by monitoring the time history of the 777.3 nm atomic oxygen line emission with two filtered radiometers mounted on the top flange of the vacuum chamber.

The spectral measurements of the O-atom beam, and target interactions have been obtained using a Princeton Instruments Optical Multichannel Analyzer (OMA). The OMA head consists of a 1024 photodiode array with a gated S-20R intensifier. The spectrograph is a Jarrel Ash 0.275m MARK X with interchangeable grating, and a maximum resolution of 3Å. The optical field of view in the chamber was restricted to a 0.090 x 0.90 cm rectangle by a collecting telescope matched to the f number of the spectrograph. Glass cut-on filters were used to prevent second and higher order spectra from being recorded.

Spectra are recorded using the OMA in the gated mode. The diode array was gated on 5 μs after the laser trigger to prevent detection of scattered breakdown radiation. A number of spectral scans of the radiation from the expanded oxygen plasma were performed over the wavelength range of 400 to 800 nm. Results were highly reproducible. The strongest signals arose from atomic oxygen emission at 777 nm. No evidence of O_2 emission was identified, however, some atomic hydrogen emission was observed due to laser ablation of the valve tip elastomers, and H_2O impurities in the oxygen feed gas.

Characterization of the beam has been also performed by ballistic pendulum measurements of the cold and hot flows. This has allowed quantization of the atomic flow rate to within a factor of two. Refinements in the larger test facility, primarily a time-of-flight mass spectrometer probe, will further reduce this uncertainty.

PRELIMINARY MATERIAL DEGRADATION STUDIES

Preliminary material degradation studies have been conducted with the small test facility. Materials irradiated have included polyethylene, Teflon®, Kapton® (untreated and oxidation resistant treated samples), Mylar®, PEEK, PBT and Carbon Epoxy composites.

Ground-based testing facilities must produce a surface morphology and mass removal rate similar to that produced on orbit to provide meaningful erosion data. Typical erosion morphology of a polymeric sample is shown in Fig. 4 (graciously provided by J.T. Visentine), for exposure levels of 5 to 9 x 10^{20} O-atom/cm^2, corresponding to ~1 week on orbit. Kapton samples irradiated in our facility to an exposure level of ~3.3 x 10^{20} O-atom/cm^2 show a remarkably similar surface morphology Fig. 5, and measurable mass loss (~1.3 mg). (Fig. 6 shows a sample of Kapton 500H prior to exposure for comparison.) This morphology is not due to kinetic energy alone since another sample of Kapton was bombarded with ~5 eV Ar (Fig. 7) and showed no mass loss. Similar results were obtained with low and high density polyethylene samples.
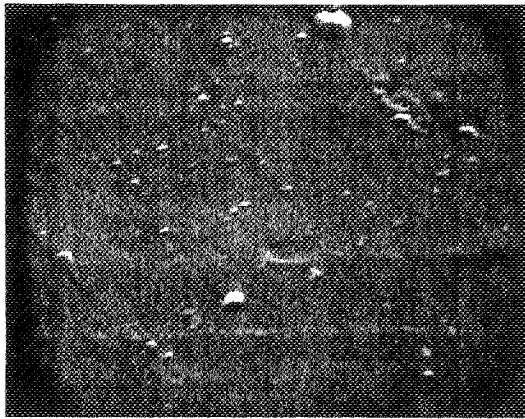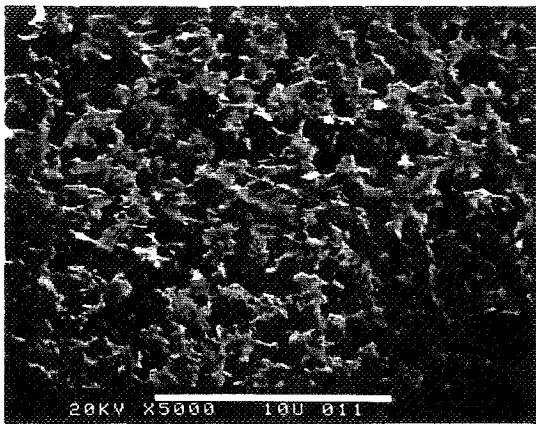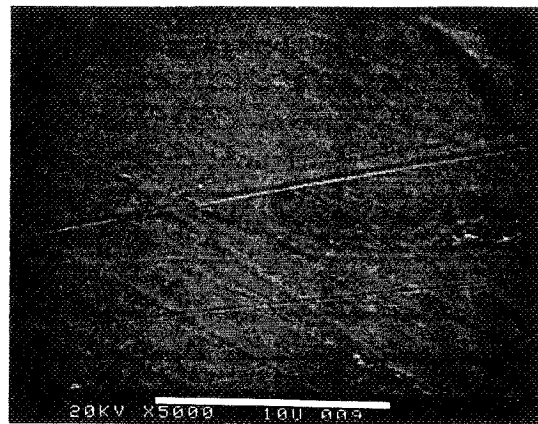
PRIOR TO EXPOSURE
(10,000x)
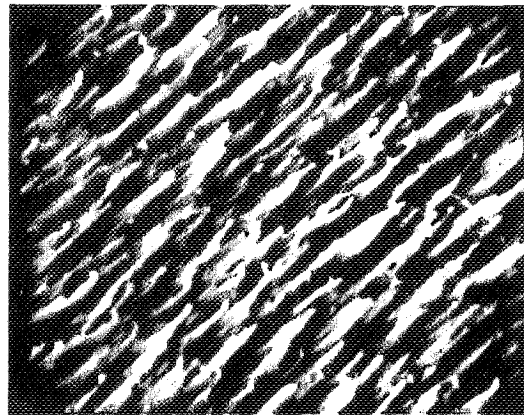
AFTER ATOMIC OXYGEN EXPOSURE
(10,000x)

Fig. 4. SEM photograms of STS-8 Kapton specimens

AFTER 12000 PULSES ($\sim 3 \times 10^{20}/cm^2$)

BEFORE EXPOSURE

Fig. 5. Kapton 500H after 12,000
pulses ($\sim 3 \times 10^{20}/cm^2$)

Fig. 6. Kapton 500H before
exposure

Fig. 7. Kapton 500H irradiated by 5 eV Ar for 12,000 pulses

Current capabilities provide for exposure rates of ~1.5 x 10^{20} atom/ cm^2-hr at ~5 eV (~1.6 Hz); and provide an exposure acceleration factor of 50 compared to LEO and 5000 for space station altitudes for ~6 cm^2 samples. A larger facility described below will allow for further accelerated testing.

DESIGN OF LARGE-SCALE TEST FACILITY

The small test facility produces high density O-atom beams, but cannot irradiate large sample areas. Using the same techniques, we are constructing a large chamber to allow the testing of larger samples (~15 cm diameter) at a 10 to 20 Hz pulse rate.

In order to accommodate larger samples, or many smaller ones, the new chamber will be 40 cm o.d. six-way cross assembly. A chamber this size allows for convenient sample access, mass spectrometer probes for beam and target product analysis, in situ mass loss measurement, velocity dependent measurements and target irradiation by a UV solar simulator. A schematic of the chamber is shown in Fig. 8.
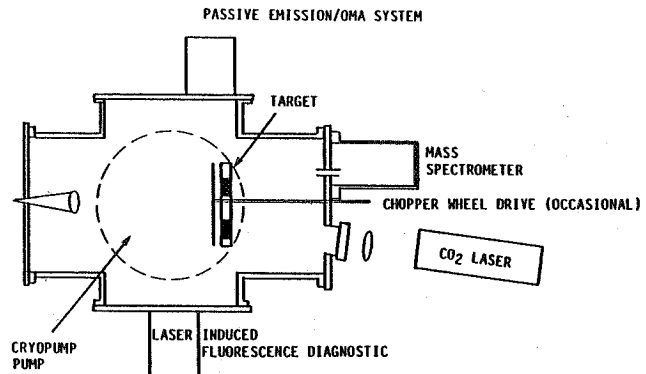

Fig. 8. Schematic of new O-atom facility

A Varian cryopump with a speed of ~3000 ℓ/s will maintain 10^{-4} torr in the chamber with a gas load of 10^{-3} g/s. A cryopump was chosen to eliminate any possible contamination of test samples by back diffusion of pump oil.

Two quadrupole mass spectrometer systems will be coupled to the main chamber. A Balzers QMS 311 will be used to monitor the atomic beam. It will sit in a differentially pumped chamber, separated from the main chamber by a sampling orifice. This QMS will monitor the pulse to pulse reproducibility of the O-atom beam, and when cross calibrated with the laser induced fluorescence diagnostic, will provide a real time quantitative O-atom detector. A second QMS will be suspended from the top flange of the chamber to monitor the reaction products of the atomic oxygen/interaction and will be mounted to allow for rotation with respect to the target for angular product dependence studies.

The pulsed CO_2 laser system was manufactured by Laser Applications Limited and provides 18J per pulse at 10 Hz. This allows a sufficient margin for transmission and reflection losses to process ~10^{-4}g bursts of gas into 5 eV O-atoms. Furthermore an option for 20 Hz operation is available to provide a higher throughput.

A Quantel 10 Hz YAG pump dye laser system will be used for multiphoton laser induced fluorescence detection of atomic oxygen. Approximately 1 mJ at 226 mm can be obtained for the excitation of ground state atomic oxygen

$(2p^4 {}^3P \rightarrow 2p^3 3p \, {}^3P)$ with subsequent fluorescence around 845 nm from the $3_p {}^3P \rightarrow 3_s {}^3S$ transitions in atomic oxygen, and the same technique can be used to detect the 1_D and 1_S metastables.

SUMMARY

Our goal is to build a reliable ground based energetic O-atom test facility which will be able to meet the NASA material testing requirements.

The concepts of the design have been demonstrated both experimentally and theoretically, and the technique exploits the characteristics of existing commercially available high power laser technology. At present repetitively pulsed CO_2 lasers provide the most convenient source of laser radiation in terms of reliability, cost and delivered energy.

With a 10 Hz pulse rate, the device will conservatively irradiate a 100 cm^2 area with $>10^{17}$ atoms/cm^2-s. In less than 2 hr of operation under these conditions, energetic oxygen atom fluences of 7×10^{20}/cm^2 will be possible over the exposed area. For comparison this O-atom fluence is equivalent to that seen by a ram direction shuttle surface for an entire week-long mission in low earth orbit (250 km) and is equivalent to the fluence seen by a ram surface of the Space Station at 500 km altitude during the course of an entire year (during a year of average solar activity). Although questions can be raised by accelerated testing, this new facility will provide the ability to identify materials most affected by O-atoms (which can then be tested further on-orbit), to systematically separate effects of UV irradiation, temperature, and O-atom energy, to study the rate of change of recession/ material loss as surfaces change character, and to identify failure points (most susceptible to erosion) in assemblies such as solar cells. The importance of such effects has been clearly demonstrated in recent Space Shuttle flights.(5, 9-11).

With this new test facility we can perform quantitative erosion testing of materials, components, and even small assemblies (such as a solar cell array) in order to determine components or interfaces which are most vulnerable to O-atom erosion. This ground-based facility will allow accelerated testing to identify structures or materials which are in most critical need of protection so that remedial strategies or protective coatings can be developed and even tested in this ground test facility at a fraction of the cost and in a much more timely manner than on-orbit.

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