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DEVELOPMENT OF A NOZZLE BEAM CONTAINING ATOMIC OXYGEN

by

Calvin K. Lam



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# SUMMARY

Initial studies of the feasibility of producing a hypersonic molecular beam containing appreciable amount of atomic oxygen have been successfully completed. The purpose of this work was to produce a molecular beam which could be used for basic and applied studies related to the interaction of the earth's upper atmosphere, such as accommodation coefficient measurements or surface reactions at high kinetic energy.

A quartz tube source was developed for the molecular beam in which a 200 watt microwave discharge could be maintained. The gas from the discharge passed through a small orifice and was expanded into a free jet from which a beam was extracted utilizing welldeveloped nozzle beam techniques. The percentage dissociation of oxygen in the beam was measured using a mass spectrometer with an "in-flight" ionizer. The studies included the effect of pressure and orifice size, for which a compromise was involved in order to achieve high Mach numbers and high dissociation levels, the use of double cavities, and the effect of small additions of  $H_20$ . It was demonstrated in these initial tests that a hypersonic beam from a 92% He 8% 0<sub>2</sub> mixture could be produced with 17% dissociation. This figure can very likely be increased substantially with further optimization.

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NOTATION

a	sonic velocity
А	orifice area
cp	specific heat at constant pressure
c <sub>v</sub>	specific heat at constant volume
đ	molecular diameter
D	orifice diameter
k	Boltzmann constant
Kn	Knudsen number
I	molecular flux per solid angle (molecules/ steradian/sec)
M <sub>f</sub>	freezing Mach number
n	number density
N	Laschmidt number = $2.74 \times 10^{19}$ molecules/c.c.
Ρ	pressure
Pa	atmospheric pressure
R <sub>s</sub>	special gas constant
Т	temperature
v	velocity
V	volume
х	distance along X-axis
γ	ratio of the specific heats, $c_p/c_v$
$egin{array}{c} \theta \\ \lambda \\ \lambda_D \end{array}$	deviation angle from X-axis mean free path Debye length
σ.	total molecular flux (molecules/sec)
Ω	solid angle (steradian)
()c	condition of the charged particles
( )col	condition in the collimation chamber and at the collimator

(	)e	condition	of	the	electrons
(	)ex	condition	in	the	expansion chamber
(	)in	condition	in	the	interaction chamber
(	) o	condition	in	the	source
(	)sk	condition	at	the	skimmer
(	)t	condition	at	the	target
(	) *	choke flow	cc	ndit	ion

# I. INTRODUCTION

Since high-energy nozzle type molecular beams were devised, much research work has become possible which could not have been done with classical oven-beams. This is especially significant when studies of gas-surface interactions and satellite aerodynamics are being considered, because beam energies of 1 to 10 eV and necessary beam intensities that duplicate the satellite flight conditions were made possible. Nozzle-beam technique has been described by a variety of authors, see, for example, Ref.1.

Nozzle beam studies to date have been limited primarily to noble gases or stable diatomic gases such as nitrogen. Time is now ripe to produce molecular beams with reactive gases, e.g., atomic oxygen and nitrogen, that will simulate more closely in the laboratory the gas-surface interactions that a satellite will really encounter in the upper atmosphere. The investigation of actual space-flight dynamical effects, chemisorption effects and associated phenomena could then be attempted.

The aim of this present program was to produce, with the help of microwave power discharge, a high-velocity nozzle-beam that contains atomic oxygen from dissociation of  $0_2$ . There were a few means available to help reaching this goal, for instance, dissociation of  $0_2$  by ohmical heating or by intermittent capacitive D.C. discharge and by titration of N with NO. The advantage of using microwave power (i.e. electrodeless) discharge on gas flowing in quartz tube as was done in this work is that it gives a very "clean" (uncontaminated) beam containing constant (against intermittent) supply of a larger amount of atomic oxygen.

Firstly ionization, then dissociation of the gas occurs under microwave discharge. The charged particles thus produced would recombine with each other very quickly; whereas the radicals can survive much longer. The reasons are that wall recombination of the atoms could be drastically reduced by using well-washed quartz containers; and that bulk recombination, in order to fulfill both conservation of energy and momentum, requires multi-body collisions which have very low frequencies. These surviving radicals, once the beam has expanded to a collisionless state, will remain in the beam as atoms without recombinations.

There are a few factors that may affect the amount and speed of dissociation, the nozzle pressure being the most dominant one. Rose and Brown (Ref.2) have shown that the optimal breakdown pressure for pure oxygen was around 4 torr with a working pressure within the range of 1 to 10 torr. According to Fenn et al (Ref.21) the freezing Mach number of the beam is proportional to the nozzle orifice diameter (D<sub>o</sub>) and source pressure (P<sub>o</sub>):

$$M_{f} = 1.8 (1/Kn_{o})^{\gamma/(\gamma-1)}$$

where

 $Kn_o = \lambda_o / D_o$ 

and  $\lambda_0 = 1/(\sqrt{2*n} * \pi * d^2)$ 

where n = 
$$2.74*10^{19} (P_760)*(273/T_0)$$

nence 
$$M_{f} = 1.8 (D_{\star} \sqrt{2} * * d^{2} * 2.7 * 10^{19} * 273 * P_{o} / 760 * T_{o})$$

Because of the low nozzle pressure required to accommodate the electric breakdown it was attempted to install a source with a large orifice to compensate for this in order to create, within the capacity of our apparatus, a useful beam intensity and Mach number. Preliminary calculations (see also Appendix A) before experiment showed that a compromise among all the limiting parameters yielded a Mach number of 5 for a pure oxygen beam. To further improve this situation, the seeding technique with helium as a light carrier gas was employed. This would help to boost the terminal velocity of the seeding gas, but may not have increased its speed ratio. As reported by Abuaf et al (Ref.3) and discussed by Anderson (Ref.4), the resultant Mach numbers (or speed ratio) of the heavier gases (0 and  $0_2$ ) may not be raised even though their terminal velocity were also increased to that corresponding to the mean molecular weight. Whatever speed ratio effect there is in the experiment would remain uncertain until the velocity distributions are measured. It was also hoped that by mixing oxygen in an inert gas the discharge could be sustained at a higher total pressure while the partial pressure of the oxygen is still within the range of the above-mentioned breakdown pressure. The freezing Mach number can then be further increased by raising the nozzle parameter.

It is desirable to know the percentage of dissociation at the discharge section and the subsequent change during beam expansion, but in these initial studies the amount of atomic oxygen in the final beam at the location where further experiments of gassurface interactions would be conducted is what we are more concerned about. A mass spectrometer mounted in the interaction chamber (Fig.l) has been relied on for this analysis.

### II. EXPERIMENTAL APPARATUS

# 2.1 The Stagnation Source Tube and Exit Nozzle of the Beam System

The nozzle-beam apparatus (called the mini-beam because of its compact design) is shown in figure 1.

The stagnation source tube and exit nozzle that is needed for accomodating the microwave power discharge is made of quartz. Quartz furnishes as clean and stable a surface as pyrex glass, which, unlike metallic surfaces, is very inefficient for recombining the gas radicals (Ref.5). The high melting point (1300°K) of the quartz provided us the option of raising the nozzle temperature and in turn the kinetic energy of the beam produced.

The stagnation source was made of 10 mm i.d. quartz tubes.

Instead of a simple straight tube it takes on a special configuration (Fig.3). Downstream of the gas bottle, the quartz tube is split for a short distance into two parallel branches before it rejoins to feed the nozzle. The electric discharge is brought about by two discharge cavities, one being coupled to each parallel branch of the quartz tube. This has been found to produce more dissociated gas than using a single cavity at an unbranched section (Ref.6). Three nozzles were prepared, one with a 0.009" orifice, the second a 0.015" and the third a 0.0292", to match a wide range of nozzle pressures.

A stainless steel flange is attached to the outside surface of the quartz tube, through a vacuum-tight quartz-to-metal joint, so that the gas passage is completely free of any metallic surface. This stainless steel flange is in turn fastened, with an O-ring-sealed interface, to a stainless steel bellows-and-flange combination (Fig.4) which allows the nozzle a one degree translational and two degrees rotational freedom of motion. These freedoms are needed for ease of aligning with the subsequent collimation devices downstream of the nozzle. The whole source unit is mounted on the body of the expansion chamber with a Viton O-ring seal.

# 2.2 The Vacuum System

Figure 2 is a photograph of, from right to left, the mechanical backing pump, the mini-beam system and the interaction chamber in which the beam characterization tests were performed. The source is mounted on the right hand side of the rectangular expansion chamber, which also contains the first conical collimator or skimmer to define the beam from the free jet. The skimmer is made of stainless steel with a 0.0575" opening and a sharp lip with internal and external half angles of  $35^{\circ}$  and  $45^{\circ}$  respectively. The internal angle and the sharpness of the lip are especially important if the degradation of the flow is to be at a minimum.

The expansion chamber for the free jet emerging from the nozzle is pumped by a NRC HS-10 oil diffusion pump with a charge of DC 705 fluid. This pump has a capacity of 2200 litres/sec for air with a baffle (ultimate pressure of the order of  $5 \times 10^{-10}$  torr) or 4200 litres/sec without a baffle (ultimate pressure of  $5 \times 10^{-8}$  torr). The second pumping stage, which removes the uncollimated beam molecules, consists of a pyrex glass cross chamber with an Edwards F 903 oil diffusion pump with a charge of DC 705 fluid. This pump has an ultimate pressure capability of  $5 \times 10^{-7}$  torr at a speed of 1500 litres/sec for air without the use of a baffle.

A Balzers IMR-6 ionization gauge is mounted in each of the two chambers for the measurement of background pressure. A second collimator with a 3/8 inch diameter orifice is mounted in the pyrex chamber to further define the beam when it enters the interaction (or target) chamber. The collimator is 13.375 inches away from the skimmer. A 2-inch isolation valve separates the beam generation system from the adjacent interaction chamber.

#### 2.3 The Interaction Chamber

The interaction chamber, made of stainless steel and electropolished on all inner surfaces, is of a very versatile design, with interchangeable flanges on all sides, and with two hinged doors for easy access as shown in Fig. 2. All the joints are sealed with viton 0-ring, which are greased with APIEZON L.

A NRC type HS-10 diffusion pump, with a liquid nitrogen baffle is mounted on the chamber. This system has a net pumping speed of 2200 litres/sec for air, with an ultimate pressure capability of about 5 x  $10^{-10}$  torr. The best available diffusion pump fluid, viz. DC 705 (made by Dow Corning), is used because of its extremely low vapour pressure and minimal backstreaming at elevated pressure.

The target chamber can be isolated from the pump stack by means of a 10 inch plate valve.

A Varian Vac ion pump with a speed of 15 litres/sec for air is mounted on the chamber in order to maintain a vacuum of better than  $10^{-6}$  torr between successive experiments. This eliminates repeated exposure of the system to the atmosphere, thus reducing the possibility of adsorption and subsequent release of gases by the system.

The system was initially designed for an ultimate pressure of  $1 \times 10^{-8}$  torr in the target chamber. A Balzers IMR-7 ionization gauge of the Bayard-Alpert type is mounted on the chamber to measure total pressure. The overall system **achievecabbaseppressure** of  $3 \times 10^{-9}$  torr.

# 2.4 Microwave Power System

The frequency of microwave power most popularly used for heating and medical purpose is 2450 M Hz. Microwave power generators of this kind are commerically available.

# 2.4.1 The Components

A MICROTRON 200 Mark II of this frequency was bought from Electro-Medical Supplies, England. Its maximum output power is 200 watts (Fig.4). The Microtron 200 incorporates a German-made Mullard type JP2-02 continuous wave Magnetron and a full-wave rectifier. The rectified D.C. is well filtered to remedy the small irregularity or surge of microwave power experienced by Burt (Ref.6). The unit has been adjusted to take supply of 110V A.C. at 60 cps. The cooling for the magnetron is blown air from a built-in fan. The machine is also equipped with an output power wattmeter.

A coaxial cable for power transmission and two discharge cavities, model No. 214L, were also procurred from the same supplier. The 214L is a cylinder approximately 1" diameter by 2-1/4" long with a variable coupling at right angle to the cavity length (Fig. 5). The quartz tube is fitted through one end of the cylinder at right angle to both the cavity length and the incident power coupling. The other end of the cylinder is blanked off, with a tuning stub mounted at the centre of the end plate longitudinally in line with the cavity axis. The coaxial input is a type-C connector integral with the coupling adjustment and two cooling air inlets. The coupling adjustment together with the tuning stub help obtaining the best match for different nozzle flow conditions. The 214L is designed to be used with the Microtron 200.

The compressed air line was provided for controlling the temperature of the discharge cavities and the quartz tube, which can get very hot at high power discharge without cooling. The compressed air was filtered through a Leslie ATMO, AFG-1 pressure reducing valve to prevent depositing of oil drops on the inner wall of the cavities.

A power splitter (magic tee), EMCO D-105N is used to equally divide the microwave power into the two discharge sections of the source. This limits the maximum incident power to 100 watts per channel. The EMCO D-105N can handle a maximum of 250 watts average power and 2.5 kilowatts peak power. The maximum input Voltage Standing Wave Ratio is 1.4. The impedence of the unit is 50 ohms.

A Tesla Coil was secured for initiating the microwave discharge.

# 2.4.2. Reflected Power Measurement

The C.W. Magnetron of the Microtron 200 can withstand only 75 watts reflected power for a short period or 40 watts maximum continuously. To monitor this, a resistive-loop directional coupler, EMCO Cll3N, was inserted between the coaxial exit connector of the machine and the transmission cable. The coupled signal was fed to a semiconductor circuit to be rectified and measured by a Hewlett Packard hp 410C voltmeter.

The EMCO C-113N is a three-port single-direction coupler with a 20 db minimum directivity and 40 db coupling. Its maximum permissible power flow is 500 watts average and 5 kw peak. The maximum insertion loss is only 0.2 db and its impedence 50 ohms.

The semiconductor circuit is a half-wave D.C. rectifier employing a IN831 Alpha single-forward type microwave diode, a 1/8 watt, 50 ohm resistor and a 5 micro-micro farad filter (Fig.6). The connection between the rectifier and the coupler was furnished by an Amphenol RG 58/U coaxial cable.

To obtain a calibration curve (Fig.7) correlating the microwave power and the rectified D.C. voltage, the directional coupler was first inserted in the same direction as the forward power (Fig.8a) which was then discharged to a gas flow through a roughly tuned cavity. With the help of the exact reading of the output power wattmeter, the forward power and the corresponding coupled signal, represented by the rectified D.C. voltage, can be plotted in a graph. In normal use, when the reflected power is being monitored, the coupler is connected in the direction of the reflected power (Fig. 8b). Observation and control was done manually to avoid too high a reflection.

This measuring circuitry enables one to tune the discharge cavity to best match a given nozzle flow condition.

# 2.5 Gas Source

The seeding technique and the different partial pressures of oxygen at microwave breakdown require different gas compositions for an optimal operating mixture.

The gas mixture that feeds the nozzle is prepared internally in our laboratory. A batch type multicomponent gas mixing system has been designed (Fig.9). Its compactness and portability goes well with the mini-beam machine. The operation of this system is included in Appendix B. The main features are as follows:

# 2.5.1 Batch Mixing

Helium gas of 99.9995% purity was obtained from Linde. The gas contains less than 2.0 ppm each of neon and nitrogen and less than 1.0 ppm each of all others. Therapy Oxygen of 99.5% purity was supplied by Ohio Chemical Canada Ltd.

The empty bottle was first filled with, say, oxygen to about 4 atmospheres then the contents were released. The bottle was "flushed" in this way a few times to make sure that the residue inside the empty bottle was mainly oxygen at atmospheric pressure.

The empty bottle, with residual oxygen at 1 atmospheric pressure (14.7 psia), was filled with oxygen to a pre-designed pressure. Helium was then used to make up the rest of the bottle to a maximum tank pressure of 500 psig, which was arbitrarily chosen for safety and convenience. A working example is listed below:

Species Pressures Introduced Successively	Partial Pressure of each Species (psia)	Total Pressure of the Bottle (psig)
Residual 02	14.7	0
Oxygen	26.0 + 14.7	26.0
Helium	475.0	501.0

The total pressure in the bottle is then 515.7 psia and the percentages by volume of each constituent are:

for  $0_2 = \frac{26.0 + 14.7}{515.7} \times 100\% = 7.89\%$ 

for He =  $\frac{475.0}{515.7}$  x 100% = 92.11%

This gas mixture was used throughout this work.

The oxygen bottle is equipped with a Millaflow balance pressure regulator 41000646. The helium bottle has a National 5700 series regulator. Both regulators are protected from back pressure by a Whitey 1KM4 shut-off value at their outlet lines.

Two 6" Conval test gauges 60-1379-2L (Gauges #1 and #2) are used for accurate reading of the pressure in the gas mixture bottle. Both gauges have mirror-ring dial surfaces. The maximum dial reading of gauge #1 is 100 psig while that of #2 is 600 psig. Gauge #1 is also protected by a Whitey 1 KM4 shut-off valve (e).

# 2.5.2 Gas Flow to Nozzle

When a gas mixture was being fed to the nozzle, a Millaflow 41000646 regulator (b) was used for the first step-down of the line pressure. Then a Nupro SS-2SA very-fine-metering needle valve (d) furnished further flow control. The nozzle pressure downstream to this needle valve was monitored by gauges #3 and #4. Gauge #4 is a 12" Heise absolute pressure gauge with maximum dial reading of 100 psia, while gauge #3 is a 6" Wallace and Tiernan absolute pressure gauge type #FA-160110 with a maximum dial reading of 50 torr with 0.2 torr divisons. This low pressure gauge was protected by a Whitey OVS2 shut-off valve in case the nozzle pressure <u>wasttoolhigh</u>. Both gauges #3 and #4 have mirror-ring dials for accuracy.

The water vapor source (m) is provided for 'wetting' the gas feeding the nozzle. The reservoir is just a simple copper tube of water. The 23.7 torr saturated water vapor at room temperature  $(77^{\circ} \text{ F})$  was let into the main stream of gas flow through a Nupro SS-2SA needle valve (n). For nozzle pressure greater than 23.7 torr, the copper tube has to be immersed into a boiling water bath to obtain sufficient pressure.

Downstream of this water reservoir the line is connected to a type 321 stainless steel bellows (p) that is joined to the quartz source at the other end through a Mason-Renshaw Compress-Ocoupling which is composed of a type 304 stainless steel body and a Viton-A O-ring.

# 2.6 The Mass Spectrometer

The mass spectrometer used is a model AVA 1 vacuum analyzer consisting of an AST 1-B tube made by the Aero Vac Corp. with the ion gun electrodes extending in the nude type of configuration into the vacuum system. The AST 1-B tube is a magnetic type single-focussing mass spectrometer (Ref.7). A total pressure in the range of  $10^{-4}$  to  $10^{-10}$  torr can be measured by the analyzer. The mass number range is in two stages, from 2 to 11 AMU, and from 12 to 72 AMU, obtained by using two different sizes of deflection

magnets. The ion accelerating potential has a range of 75V to 400V, and can be set manually at any value within this range or left scanning through the range automatically with repetition cycles of 2 minutes or 1/2 minute duration.

The partial pressure signal (ion current) from the spectrometer was fed to a Keithley 640 vibrating capacitor electrometer which can measure currents as low as  $10^{-14}$  amp. The amplified output signal from the Keithley was recorded by a Mosley Autograf model 7030AM X-Y recorder.

The spectrometer was aligned with the molecular beam system by optical means using a laser and a telescope. The molecular beam can be flagged and unflagged from the spectrometer by a plate mounted on a movable arm extending out of the vacuum chamber through a flexible bellows.

# III. EXPERIMENTAL RESULTS

# 3.1 Operation of Mini-Beam System

When the source was mounted on the mini-beam machine it was aligned with the skimmer, the collimator and the mass spectrometer by means of a telescope. The mass spectrometer tube was removed from the interaction chamber. A telescope was set up to look at the collimator and the skimmer openings through the mounting hole of the mass spectrometer tube. At the other end of the machine a lamp was used to shine light through the nozzle. By bringing in successively the centers of the mass spectrometer mounting hole, the collimator opening, the skimmer opening and the nozzle orifice as close to the cross-hair of the telescope as possible, the alignment was completed.

With the freedom of motion of the nozzle provided by the bellows, the nozzle orifice could be set right at the centre of the skimmer. Nevertheless, due to imperfections in fabrication and assembly of the system, the centre of the mass spectrometer tube was out of alignment by 1/8". This gave only a very small angle of misalignment, namely,  $5.79 \times 10^{-4}$  radians. According to Sherman's formula (Ref.8) for a free jet expansion:

 $n_{(x,\theta)} = .161 n_{0} (X/D)^{-2} \cos^{2} 1.151\theta$ 

the effect of this angle in the  $(\cos^2 1.151\theta)$  term is negligible and  $n_{(x,\theta)}$  is practically equal to  $n_{(x,0)}$  along the centre line.

Included in Appendix A is a detailed theoretical model calculation of the free jet expansion and the flux and pressure in different chambers of the machine with a nozzle temperature and pressure at  $77^{\circ}$ F and 5 torr respectively. The gas mixture is 7.89% O<sub>2</sub> and 92.1\% He, which was used all through this work. The orifice size of the nozzle used in the calculation was 0.029".

The experimentally recorded pressures of the chambers

chambers	expansion	collimation	interaction
pressures	chamber	chamber	chamber
calculated chamber pressure (Appendix A)	$2.4 \times 10^{-4}$ torr	6.28 x 10 <sup>-7</sup> torr	$1.7 \times 10^{-7}$ torr
Balzer IMR gauges	$2.08 \times 10^{-4}$	1.22 x 10 <sup>-6</sup>	$3.71 \times 10^{-7}$ torr
recorded pressure	torr	torr	

are listed below together with the ones calculated:

The IMR gauge recorded values were all adjusted with the gauge factors associated with the appropriate gas mixture in different chambers. The ideal flow values calculated in Appendix A are perhaps lower than the actual values because of the departures from ideal behaviour during the expansion. From the above table, it can be seen that the recorded pressures in the collimation chamber and interaction chamber are higher than the calculated ones. This suggests that more gas may have issued from the nozzle than was calculated. However, this trend was reversed in the expansion chamber. This reversal may have been caused by the fact that the bulk of the light carrier gas is being pumped at this stage. The light gas in turn causes a higher actual pumping speed than the rated one used in the calculation. The effect diminished in the subsequent chambers because the percentage of light gas in the flow decreased after the skimming process.

The calculated flow rate of the nozzle orifice is 1.16 atmospheric c.c. per second, and the calculated freezing Mach number is 4.66. This Mach number is equivalent to a beam speed of 1587 meter/sec which is only 1/4 of that of a typical satellite orbital velocity. The beam energy then is 1 eV for 0<sub>2</sub> and 0.5 eV for 0.

The total number of molecules arriving at the mass spectrometer is calculated to be  $9.12 \times 10^{14}$  molecules/cm<sup>2</sup>/sec. There was no effort made to cross-check this figure with a calibrated mass spectrometer.

The enrichment effect at the target of the heavier species from the seeding technique was also investigated. Having adjusted the peak heights on a mass spectrograph with appropriate ionization cross-sections, 2.55 x  $10^{-16}$  cm<sup>2</sup> for  $0_2$ ; 0.38 x  $10^{-16}$  cm<sup>2</sup> for He (Ref.7) and  $1.581 \times 10^{-16}$  cm<sup>2</sup> for 0 (Ref. 9). A maximum of 37.1% of  $0_2$  was deduced. Since the sensitivity of the mass spectrometer towards different gases was not calibrated, it was taken as the same for all species in the above deduction. The gas mixture used was 7.89%  $0_2$  and 92.1% He while the nozzle pressure and nozzle orifice diameter were 47.75 torr and 0.009" respectively. This enrichment is comparable to that reported by Klingelhöfer and Lohse (Ref.10), 40% Ar from a 1% Ar--99\% H<sub>2</sub> source mixtures; and that reported by O'Keefe (Ref.11), 9.6% Ar from a 1% Ar--99% He source. This seeding technique has boosted the number of  $0_2$  molecules arriving at the target to  $3.6 \times 10^{14}$  molecules/cm<sup>2</sup>/sec.

# 3.2 Mass Spectrometry

The mechanism of dissociation and recombination at the glow-discharge section of the source is quite complicated. There was no attempt to investigate this aspect of the problem. For the purpose of the studies of gas-surface interaction, it suffices at this stage to record the relative abundance of atomic oxygen and molecular oxygen in the beam at the location of the mass spectrometer tube where, in future, targets to study gas-surface interactions will be mounted.

# 3.2.1 Operating the Mass Spectrometer for "In-Flight" Measurement

There are a few differences between this fly-through mode of measurement of mass spectrometric analysis for molecular beams and the usual stagnant chamber background gas analysis.

Firstly, the high directivity of the molecular beam is totally different from the isotropical character of velocity of a stagnant gas. The directed velocity of 1587 meter/sec at Mach number 4.66 (Appendix A) is much greater than the arithmetic mean velocity of 622 meter/sec at  $77^{\circ}F$  for a stagnant gas of the same composition.

Secondly, the intensity of the molecular beam that feeds the mass spectrometer tube cannot be directly related to the background pressure. For example, a beam from a 5 torr nozzle pressure (Appendix A) generates a beam intensity of 9.12 x  $10^{14}$  molecules/ cm<sup>2</sup>/sec that strikes the target. The beam exit velocity from the source nozzle orifice is 80450 cm/sec therefore, the number density of the beam arriving the target is 1.13 x  $10^{10}$  molecules/c.c. A stagnant system of gas at this same background pressure and room temperature represents a number density of 1.33 x  $10^{10}$  molecules/c.c., about the same as the directed beam.

In this work, all the mass spectrographs were recorded with the accelerating potential scanning at the two-minute cycle. Because of the above mentioned characteristics of a directed beam, it was found that for this fly-through mode of measurement of beam compositions the accelerating potential had to be left in service continuously so that identical mass spectrographs could be reproduced when source conditions were the same. Meaningful analysis was thus made possible. Every time after new conditions were set upstream of the mass spectrometer, the accelerating potential was allowed to scan through the mass spectrum for two free runs before a useful spectrograph was recorded.

The reason behind this way of operation may be accounted for by the molecules being adsorbed to the surfaces of the metallic parts of the mass spectrometer at the proximity of the entrance slit. These molecules, having been adsorbed in the past history when the accelerating potential was off, could be knocked down by the accelerated ion stream when the potential is on; and could induce a false or unsteady signal. An equilibrium between these absorption and desorption could only be achieved with the scanning potential in continuous service.

# 3.2.2 The Effect of Flagging the Beam

Mass spectrographs were produced with the beam flagged and unflagged for the same upstream conditions. When the beam was flagged the peak height of each mass number was generally less than half of that recorded when the beam was unflagged. The reason for this has been pointed out in section 3.2.1., because the mass spectrometer was measuring the background molecules when the beam was flagged while the record was for both background and beam molecules at an unflagged position. Here the number densities of both background and the beam are approximately the same.

The relative abundance for a flagged beam were usually identical when microwave power was turned on and off. This suggested that the stainless steel flag and the wall of the interaction chamber had very effectively helped recombine all the atomic oxygen produced by the glow-discharge (Ref.5).

The peaks in the spectrographs were narrow so that the peak heights gave very good indications of the abundance of each species. Therefore only the peak heights instead of the integral area under the curve at each mass number were used for subsequent analysis.

# 3.2.2 Interpretation of the Mass Spectrograph

It is impossible to separate a recorded peak height, say, of mass number 16, in a mass spectrograph (Fig.10), into the fractions that are contributed by different species.  $0^{++}_2$  is one possible constituent;  $0^+$  should make up the rest, among which some would be from cracking down (dissociative ionization) of  $0_2$  and  $H_20$  by the electron gun of the mass spectrometer while the others would be genuine atomic oxygen from dissociation by the glow-discharge at the source.

To avoid the above complication, the analysis in this work was done in the following steps. First, one compares the peak heights of the mass numbers 16 and 32 on one spectrograph recorded from an unflagged beam with no microwave power discharge at the source. This yields one  $0/0_2$  ratio. Then one turns on the microwave and records again the  $0/0_2$  ratio for the same nozzle flow conditions. Subtracting the first  $0/0_2$  ratio from the second figure, one obtains an indication of the increase in oxygen atoms resulted from the incident microwave power.

The test results of all the twenty-one runs are listed in Table I, where all spectrographs are from unflagged beams.

# 3.2.4 Dissociative Ionization of a Directed Beam

When  $0_2$  and  $H_20$  molecules are being ionized by the electron gun of the mass spectrometer, some of the molecules are being cracked down to 0 and 0H radicals which in turn may or may not be ionized at the same time.

 $0_{2} + e \rightarrow 0^{+} + 0 + 2e$   $0 + e \rightarrow 0^{+} + 2e$   $H_{2}0 + e \rightarrow 0^{+} + 0H + 2e$   $0H + e \rightarrow 0H^{+} + 2e$ 

It was found that the mono-energetic molecular beam gives a different mass spectrograph than that of a stagnant system of the same composition. The beam could be flagged from the mass spectrometer. When this was done the beam was scattered by the flag. The molecules then diffused and entered the mass spectrometer isotropically, since they formed a stagnant system with a Maxwellian distribution. The mass spectrographs recorded were very different from those with the beam unflagged. The  $0/0_2$  ratio was always higher for a flagged beam. Some recorded values are listed below:

Pressure	(torr)	28.1	47.75	83.8	3.4
Flow Rate	(Atm. c.c./sec)	.623	1.062	1.861	
0/02	Flagged	.755	.719	.686	
Ratio	Unflagged	.614	.611	.573	

The cause is thought to be due to the directivity of the beam and the crackdown pattern of the oxygen molecules. As was reported by Dunn and Keiffer (Ref.12) the majority of the radicals from the break-up of  $H_2$  had a tendency to fly apart parallel or anti-parallel to the direction of the electron stream. If the same was true for  $O_2$ , then when this effect is coupled with the high velocity of the beam, the resultant motion of both dissociated oxygen atoms would have made it difficult for them to be collected through the entrance slit of the mass spectrometer. On the other hand the collection of ions formed by cracking the diffuse back-ground gas cannot be affected by this directional argument because of the isotropic directional distribution therefore yielding a higher  $0/O_2$  ratio.

The same phenomenon was observed with a 'wetted' gas source, but the difference in  $0/0_2$  ratio between a flagged and unflagged beam was less prominent. Presumably this is due to some difference in the kineticsinvolved in cracking the H<sub>2</sub>O molecules.

#### 3.3 Microwave Power Discharge

# 3.3.1 Tuning and Matching

The reflected power measuring device worked well. It was in service whenever the microwave power was turned on. A calibration curve was produced covering microwave power from 35 W to 75 W(Fig.7). Either the IN831 diode, the EMCO Cl13N or the EMCO D-105N was very sensitive to ambient temperature, so that the whole calibrated curve readily shifted up when the environment became warm. Calibrations at different ambient temperatures were tried but they were found unnecessary because the resonant cavities, once tuned, did not change their match with changing temperature and the same was the case for the reflected power.

The calibration curve helped to tune the cavities to their best positions, where the reflected power was much below 20 watts according to the extended section of the curve. It was found that the cavities could best be tuned one at a time before the power splitter was employed in the circuit. After being hooked up with the power divider the separately tuned resonant cavities needed only small adjustment at the tuning stub to balance the apparent power consumption per channel. The balancing could be visually achieved by observing the brightness of the glow-discharge in both cavities. This small adjustment did not significantly increase the reflected power. Adjustment in one cavity was readily reflected in the other, typically in the form of a change in the intensity of the glow.

The match of the cavities was not critically affected when the nozzle pressure was changed a few torrs up or down. This saved the effort of retuning for each set of nozzle conditions. However, when a new source tube was put in place the cavities were retuned to get a better match.

# 3.3.2 Incident Power Versus 0/0, Ratio

Experimental results were presented in Fig. 11, for 5 torr nozzle pressure and a 0.1011  $H_2O/O_2$  ratio; 4 torr and a 0.1273 ratio, and 3.2 torr and a 0.1578 ratio. These curves conformed with the general trend of a glow-discharge reported by Spindler (Ref.6) with similar microwave equipments and flow conditions. His curves showed a positive slope from 0 to 100 watts input and rather quickly leveled off beyond 100 watts. This means that incident power of more than 100 watts to a cavity did not help much to increase the dissociation. He was using a single resonant cavity. The plottings of dissociation versus power input per channel in Fig. 11 are very similar to what was reported as is shown by the slopes of the 5-torr and 3,2-torr curves and the horizontal points at 4-torr nozzle condition. No test was conducted at total input power of higher than 150 watts (i.e., 75 watts/channel) for fear of overstressing the machine. The crossing-point of the 5-torr curve is at 62.5 watts and that of the 3.2-torr curve at 27.5 watts. These seem to suggest a cut-off point where the rates of dissociation and recombination equalled each other and thus no atomic oxygen could come out of the nozzle.

# 3.4 Operational Characteristics of the Quartz Nozzles

The source tubes with 0.009" and 0.015" orifice were washed with distilled water, alcohol and again distilled water. The 0.029" one was cleaned with aqueous nitric acid and detergent, then with dilute hydrofluoric acid and rinsed repeatedly with distilled water. In all, twenty-one runs were performed with these nozzles. Breakage of the 0.015" limited the number of tests conducted with it.

# 3.4.1 Nozzle Pressure and the Percentage of Dissociation

All the three quartz source tubes were tried on the machine, which permitted nozzle pressures ranging from 3 to 215 torr with reasonable flow rates (about 0.7 to 4.7 atm. c.c./sec). There were two considerations in designing the nozzles, namely, the freezing Mach number and the percentage of dissociation. If To keep the mass flow rate out of the nozzle fixed, a high nozzle pressure would result a high freezing Mach number while a high percentage of dissociation may call for a low source pressure. Since the pumping capacity is fixed, the variation of the orifice size then determines the source pressure, the Mach number and the dissociation.

Most of the tests were done on the 0.029" nozzle which was designed for a flow rate of about 1 atm. c.c./sec at a pressure of 4 torr. This was the optimal pressure for discharge on pure  $0_2$  reported by Rose and Brown (Ref.2). Now working with a gas mixture, it was hoped that a partial pressure of  $0_2$  at 4 torr would yield the optimal effect. If this were the case a total pressure of 50.6 torr in the nozzle for our mixture of 7.89%  $0_2$  and 92.11% He would be best. This pressure would in turn produce a freezing Mach number of 11.4, a very desirable molecular velocity for subsequent experiment dealing with satellite flight dynamics and gassurface interactions.

The 0.009" and 0.015" nozzles disproved this hope. The operational pressure for the 0.009" nozzle in particular was found to be too high to permit a significant amount of dissociation. Though McCarthy (Ref.13) managed to raise the pressure for glow-discharge to greater than atmospheric and though at one point discharge was sustained at a pressure as high as 116.5 torr in the 0.009" nozzle with a mixture of 23.3%  $0_2$  and 76.7% He, yet the increase in the  $0/0_2$  ratio was negligibly small at high nozzle pressure.

Figure 12 shows the results obtained with the 0.009" nozzle. It is clearly evident that there was more dissociation at lower nozzle pressure, and the 0.12 of 0/0 ratio corresponding to a 5 torr nozzle pressure on the graph is close to the value obtained with the same nozzle pressure in the 0.029" nozzle. Results used in this figure were from tests employing a 'dry' gas source.

The results in Table I indicated that among the tests the one with a 3.2 torr total pressure (i.e. 0.256 torr partial pressure for  $0_2$ ) in the 0.029" nozzle containing 0.158  $H_20/0_2$  ratio yield the highest  $0/0_2$  ratio, namely, 0.1732.

# 3.4.2 The Effect of Using Two Cavities

Experiments were conducted using either one discharge cavity or two. When only one cavity was used, it could be hooked to one arm of the branched section of the quartz tube (side position) or to the straight portion upstream to the branches (central position). The hook-up at the central position of course included extra area of the quartz wall downstream to the glow-discharge that might increase recombination of the dissociated gas. However, Schiff and Morgan (Ref.5) measured a recombination coefficient of  $4.0 \times 10^{-5}$ sec<sup>-1</sup> for quartz which is over  $10^4$  times less than that, say, for copper; and recorded a maximum of only 25% loss of dissociated nitrogen atoms (Ref.14) by passing the flow after discharge through loose Pyrex glass wool--a much larger surface area. Therefore the effect of surface recombination due to the extra quartz wall was neglected when comparing results with other ways of hook-up, i.e., side position and twin cavities.

Runs No.8 through No.12 furnish very good comparison. The nozzle flow conditions were all identical in these tests. The only change besides the discharge cavities was the amount of cooling air. A general trend of higher increase of  $0/0_2$  ratio was observed in tests No's. 11 and 12 which incorporated two cavities, but the improvement of an extra cavity was less than the doubling of the percentage of dissociation.

It was suggested that the increase in dissociation with two cavities was due to the skin depth that the microwave could penetrate into the bulk of the plasma flow at the discharge section. The magnitude of the skin depth is of the order of the Debye length,  $\lambda_{\rm D}$ , of the plasma and

$$\lambda_{\rm D} = 6.9 \sqrt{T_{\rm e}/n_{\rm c}} (\rm c.g.s.)$$

For a model calculation of the Debye length it is assumed here to have charged particles of one sign only, namely, the electrons. Since the values of  $T_e$  and n were not measured in the tests, in order to get an idea of the magnitude of  $\lambda_D$  it was proceeded to deduce from other similar works in plasma a limit for each quantity that it can ultimately reach. The electron temperature,  $T_e$ , was put to equal to 1,000,000°K (approx. 86 eV), an unlikely high value. The number density of the charged particles, n, was assumed to be  $10^{-6}$  times of the neutrals in the region, a seemingly lower limit. This yielded a value of 1.48 x  $10^{11}$  electrons for a five torr source. The above figures amount to a maximum

$$\lambda_{\rm D} = 6.9 \sqrt{10^6/1.48 \times 10^{11}} = 6.9 \sqrt{6.75 \times 10^{-3}} = 1.8 \times 10^{-2} \text{ cm}$$

Any lower value for T and higher value for  $n_{\rm c}$  will result in an even smaller  $\lambda_{\rm D}$ . The small magnitude of the Debye length suggest that the dissociation effect of the microwave was mainly confined to a very thin sheath next to the quartz wall. To use two discharge cavities could have increased this area and in turn

## produced more atomic oxygen.

Tests No's. 8 and 10 (side position) have higher yield than test No.9 (central position). The difference might have been caused by the water vapour which, as speculated by Kaufman (Ref.16), though helped the dissociation at the discharge section also helped the recombination downstream. Recombination of this kind goes on longer in test No.9.

# 3.4.3 On the Wall Temperature of the Source Tube

The compressed air line provided to cool the discharge section was controlled by a shut-off valve. No device was prepared to measure the wall temperature of the quartz tube. The relative amount of cooling employed, as indicated in Table I, were designations of the progressive degree of opening of the control valve orifice. Since the glow-discharge is a 'non-thermal' discharge by nature, negligible amount of heat was generated by the electronically excited gas. The wall temperature of the **Source** was chiefly affected by heat transferred from the cavities, which were warmed up by the standing microwave. There are two cooling air inlets at each cavity, one mainly to cool the quartz tube and the other the power connector and the cavity body. For tests No's. 1 to 5, that is for the 0.009" nozzle, compressed air was supplied only to the first inlet, while for the rest of the experiments both inlets were connected.

The different degrees of cooling listed in Table I were all considered sufficient in the sense that the temperature of the quartz was low enough to allow no significant amount of heat to be transferred to the gas flow. If this was not the case, the pressure of the constant-mass flow in the quartz tube would have been raised due to the heat. In fact, no rise in pressure was observed. Nevertheless, the four different degree of cooling had left the quartz at distinguishable temperatures.

Though Shaw (Ref.15) found that in a flow of pure  $H_2$  at 3.13 atm.c.c./sec the yield of hydrogen atoms decreased about sixfold when the temperature of the tube was increased from  $60^{\circ}C$  to  $350^{\circ}C$ , the effect in this work with  $O_2$  was not conclusive. A higher increase in the  $0/O_2$  ratio was registered in runs No's. 4,7 and 12 compared to No's. 3, 6 and 11 respectively; whereas a reverse trend was recorded in test No's. 1, 8 and 14 which were supplied with less cooling air than No's. 2, 10 and 15 respectively.

3.5 The Effect of H20 Vapour in the Percentage of Dissociation

Water vapour was allowed into the gas mixture by means of the stainless steel needle valve ('n' in Fig.9) at the reservoir. The way of controlling this was simply adjusting the needle valve orifice for a few turns of opening; while the amount of water vapour was measured downstream as a  $H_20/0_2$  ratio on mass spectrographs recorded with flagged beams and zero incident microwave power.

A curve was plotted (Fig.13) for a 4 torr nozzle pressure with different water content. The cross-point at the ordinate for a zero  $H_20/0_2$  ratio was obtained from Fig.12 at the adjusted nozzle pressure of 42.25 torr that would turn out an equivalent mass flow rate for the 0.009" nozzle. From Fig. 13 it is evident that there exists a critical amount of water vapour for each nozzle condition which will result in the best percentage dissociation.

Figure 14 represents another set of curves which correlated the  $0/0_2$  ratio in the beam for the same absolute amount of  $H_20$  vapour under different nozzle pressures. Since the needle valve (n) orifice was choked when the nozzle pressure remained less than half the water vapour pressure in the reservoir, a fixed orifice opening, say, 3 turns, would introduce the same amount of water into the flow regardless of a small change in the nozzle pressure. Both of the curves show that under the operating conditions adopted, the percentage of dissociation was improved by a higher content of  $H_0$  vapour.

Kaufman (Ref.16) detected a 50% dissociation by discharge on saturated pure  $0_2$  (about 3%  $H_20$ ). The exact percentage of dissociation at the discharge section in this work, however, was not measured, but the mass-spectrometrically detected  $0/0_2$ ratio in the beam has a maximum of 0.1732 among all the runs. The absolute percentage of dissociation at the discharge section implied by this ratio is much below the 50% cited above, which could presumably be due either to bulk or wall recombination before the gas entered the nozzle, or due to recombination during expansion.

# IV. CONCLUSIONS

The production of a nozzle-beam which contains significant atomic oxygen was proved feasible. The 17.32% dissociation achieved in test No.20 was the best obtained. Because of the many controlling factors in the experiment, more systematic and thorough studies to determine the most favourable conditions are required. The figures representing the resultant dissociation were the  $0/0_2$  ratio. A lower ratio for a beam generated at higher nozzle pressure might contain a larger absolute number of oxygen atoms than a higher ratio for a low pressure beam. Within our working conditions a higher percentage of dissociation is more desirable than a higher absolute number of radicals; higher  $0/0_2$ ratio makes mass spectrometric detection much more accurate.

The addition of water vapour was clearly a useful way to increase the amount of dissociation but the presence of water vapour in the beam may pose complications in future gas-surface interactions. In much other similar research work the  $H_2O$  content is most unwelcome. Deckers (Ref.17) noticed that the addition of a small quantity of  $N_2$  had a similar effect. The substitution of  $H_2O$  for  $N_2$  needs further investigation.

Modification is underway to place one discharge cavity at the very entrance of the nozzle. This is aimed at cutting down the collision and wall recombinations. Kaufman (Ref.16) has suggested that the water vapour in the flow, when aiding dissociation, helped bulk recombination too. To dissociate the gas right before it expands to a collision-free stream, the new location of the discharge cavity hopefully would diminish this effect. Microwave antenna of the type C produced by Raytheon also constitutes another possibility. Although it has lower efficiency, it could be mounted downstream of the nozzle exit at the location of the free jet expansion.

It is desirable to achieve higher Mach numbers and beam intensities. This can only be brought about by higher nozzle pressures. On the one hand, a nozzle pressure below two torr is deemed impracticable for our purpose. On the other hand, there were indications that a lower pressure yields higher dissociation. One thing that can be tried then is to prepare mixtures with different  $0_2$  content. A 1%  $0_2$  mixture might permit more complete breakup of the molecule; whereas a 30%  $0_2$  mixture at low nozzle pressure might produce higher absolute number of 0 molecules at a reasonable percentage dissociation.

Charged particles and excited species certainly exist in the beam; these were detected in small quantity by Elias et al (Ref. 18). These particles did not interfere with the present work, but in subsequent experiments using this beam for other studies, their effect must be considered.

Feasibility of producing a significant dissociated  $0_2$  hypersonic beam is established. This would, in general, supplement the neutral beam and the ion beam in a variety of studies; and the production of a beam with atomic gases is, in particular, a useful step towards eventual complete simulation of the earth's upper atmosphere.

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# APPENDIX A

Model calculations concerning the free-jet expansion of the nozzle beam in the Mini-beam machine.

gas	mixture		sou	arce cond	itions			
02	He	То		Po	D <sub>0</sub> = 4	D*	A o	
7.9%	92.1%	298°K	537°R	5 torr	.029"	.075cm	.004415	cm <sup>2</sup>

(1) Total flux of molecules,  $\sigma_{ex}$ , issues out of source tube nozzle orifice per unit time.  $(\gamma+1)/2(\gamma-1)$ 

$$\sigma_{ex} = (n^*)x(a^*)x(A^*) = (2/\gamma + 1) \qquad x n_0 x a_0 x A_0 \qquad (Ref. 19)$$

where

 $a_{o} = (\gamma \times R_{s} \times T_{o})^{1/2}$ 

The apparent special gas constant, R , and the apparent ratio of the specific heats,  $\gamma$  , are:

 $R_s = 1545/(4 \times 0.921 + 32 \times 0.079) = 2476 \text{ ft} - 1b_f/1b_m - R_s$ 

 $\gamma = (0.921 \times c_{p,He} + 0.079 \times c_{p,0_0})/(.921 \times c_{v,He} + .079 \times c_{v,0_0})$ 

 $= (.921 \times .1244 + .079 \times .2193)/(.921 \times .0747 + .079 \times .1572)$ 

= 1.624 (values of constants from Ref. 20)

.. a = 2639 ft/sec = 80450 cm/sec

also

n = N x (273/293) x (5/760) = 1.65 x  $10^{17}$  molecules/c.c.

Hence,

 $\sigma_{ex} = 3.16 \times 10^{19} \text{ molecules/sec}$ 

= 1.164 atmospheric c.c/sec

=  $1.008 \times 10^3$  (µHg)-litre/sec

(2) Pressure in the Expansion Chamber (assuming all molecules in the beam are being pumped away at this stage, neglecting the small fraction that passes through the skimmer into the subsequent chambers):

Diffusion pump capacity = 4200 litre/sec for air  $P = 1.008 \times 10^3/4200 = 0.24 \mu Hg = 2.4 \times 10^{-4} torr$ (3) Freezing Mach number of the jet  $M_{f} = 1.18 (Kn) - \gamma/(\gamma-1)$ (Ref.21) where the Knudsen number is  $Kn = \lambda / D = \lambda / 0.075$  $\lambda_{0} = 1/(n \times d^{2} \times \pi \times \sqrt{2})$ while here the average molecular diameter, d, is:  $d = 2(1.1 \times 10^{-8} \times 0.921 + 1.9 \times 10^{-8} \times 0.079)$ =  $2.328 \times 10^{-8}$  cm (values from Ref. 22) :  $\lambda_{0} = 1/472.5 \text{ cm}$ Hence,  $M_{e} = 4.66$ From Ref. 23 when  $p/p_0 = 2.3 \times 10^{-4}/5 = 4.8 \times 10^{-5}$  $M^* = 1.975$ This represents a molecular beam velocity of,  $v = (M^*) x (a) = 1.975 x a = 1587 metre/sec$ which is only a quarter of the typical satellite orbital velocity. If there is no slip between the He and oxygen molecules and all the particles take on the sonic velocity of the He gas, namely, a =  $(1.67 \times 389 \times 32.2 \times 537)^{1/2}$  = 101400 cm/sec then  $v_{He} = 1.975 \times a_{O,He} = 2002 \text{ metre/sec}$ This value is an upper limit of the beam velocity under these source conditions. (4) Flux of molecules passes through the skimmer into the subsequent Collimation Chamber  $I_{ex} = 0.6276 \times \sigma_{ex}$  molecules/steradians/sec and  $D_{sk} = 0.0575"$ 

The distance of the skimmer from the nozzle orifice is

 $X_{ck} = 3.35$  cm = 1.32 inch

Therefore the solid angle,  $\Omega_{sk}^{\alpha}$ , at the nozzle exit subtended by the skimmer opening is:

 $\Omega_{sk} = \left[\pi \times (.0575)^2\right] / \left[4 \times (1.32)^2\right] = 1.494 \times 10^{-3} \text{ steradians}$ Hence, the flux into the Collimation Chamber is:

$$\sigma_{col} = I_{ex} \times \Omega_{sk} = 2.956 \times 10^{16} \text{ molecules/sec}$$

= 
$$0.943$$
 (  $\mu$ Hg)-litre/sec

(5) Pressure in the Collimation Chamber

Diffusion pump capacity = 1500 litre/sec for air

$$P_{col} = 0.943/1500 = 6.28 \times 10^{-1} \text{ torr}$$

(6) Flux into the Interaction Chamber

$$A_{col} = \pi x (D_{col})^2 / 4 = \pi x (3/8)^2 / 4 in^2$$

and

$$X_{col} = 13.625$$
 in

therefore,

 $\Omega_{col} = 5.95 \times 10^{-4}$  steradian

Hence,

 $\sigma = I \times \Omega = 1.177 \times 10^{16}$  molecules/sec in ex col

=  $.3751 (\mu Hg)$ -litre/sec.

(7) Pressure in the Interaction Chamber.

Diffusion Pump (with baffle) capacity = 2200 litre/sec for air

$$P_{in} = 0.3751/2200 = 1.705 \times 10^{-7} \text{ torr}$$

(8) Flux striking the mass spectrometer (the target)

The distance of the target from the nozzle orifice is:

The solid angle at the nozzle exit subtended by a 1 cm<sup>2</sup> target area is:

$$\Omega_{+} = 1/(147.5)^2 = 4.645 \times 10^{-5}$$
 steradian

Hence,

 $\sigma_t = I \times \Omega_t = 9.12 \times 10^{14} \text{ molecules/cm}^2/\text{sec.}$ 

#### APPENDIX B.

Description:

This is a portable batch mixing system for gas sources that go with the portable minibeam machine; this system furnishes the flexibility in the laboratory of preparing gas mixtures of different compositions and percentages.

The system consists of two general areas:

- A the batch mixing area (see Fig.9)
- $\underline{B}$  the area of controlled gas flow to the nozzle of the minibeam m/c.

# Operation Guide:

- (1) Gas flow to nozzle of minibeam machine: (route shown by solid lined arrows)
  - (i) close the shut-off valve <u>a</u>. This would allow us to isolate the area A from interferring with the gas flow.
  - (ii) If pressure in the gas mixture bottle <u>D</u> is greater than 100 psig close the shut-off valve <u>e</u> to protect pressure gauge #1.
  - (iii) If internal pressure of <u>D</u> is greater than 600 psig detach the line <u>h</u> from the flow system and cap off the connector. This is for protection of gauge #2.
    - (iv) If the desired nozzle pressure is greater than one atmospheric pressure, close shut-off value  $\underline{f}$  to protect low pressure gauge #3.
    - (v) Open shut-off valve g.
    - (vi) Internal pressure of <u>D</u> will be shown on the high pressure gauge on the regulator <u>b</u>, but will be more accurately shown by test gauges #1 or #2.
- (vii) Set output pressure of b to desired value.
- (viii) Set output pressure of pressure reducing valve <u>c</u> to desired value.
  - (ix) Adjust needle valve <u>d</u> to achieve desired nozzle pressure that would be shown on gauges #3 or #4, whatever the case is.
- (2) Batch Mixing of Gases: (route shown by broken-lined arrows).
  - (i) Completely close the regulator <u>b</u> to allow no gas downstream. The regulator serves as a shut-off valve

to isolate area  $\underline{B}$  from interferring with the preparation of a new bottle of gas mixture.

- (ii) An empty bottle (D) carrying residual He at atmospheric pressure P<sub>a</sub> (after being "flushed" a few times with pure He) is put in position.
- (iii) In area <u>A</u> one can connect as many supply bottles of different gases as he wishes (5 bottles are shown in the sketch).
- (iv) Gas will be let out from the supply bottles, <u>one at a</u> <u>time</u>, to feed the mixing bottle <u>D</u>\*\*. The percentage (by weight or by volume) of each gas ingredient could be controlled by the partial pressure of this gas that is being fed into the bottle <u>D</u>. This can best be illustrated by the following example:

For a mixture of  $0_2$ , Ar and He, first set the outlet pressure of the regulator on the  $0_2$  supply bottle at, say, 20 psig and feed  $0_2$  into <u>D</u>. Pressure inside the gas mixing bottle will rise until it reaches 20 psig. Then shut-off the  $0_2$  supply. Next, set the Ar supply to, say 50 psig and feed the bottle <u>D</u> as before. Lastly, set the He supply to, say, 500 psig and **repeat** the filling procedure.

Now this results in a bottle containing a mixture with partial pressures of 20 psia, 30 psia and  $(450 + P_a)$  psia for  $0_2$ , Ar and He respectively. The total pressure inside <u>D</u> is 500 psig. The percentages by volume are approximately (depending on the initial pressure inside the "empty" bottle <u>D</u> before any gas was filled) 4% for  $0_2$ , 6% for Ar and 90% for He. \*\*see (viii)ff for detailed method.

- (v) Close shut-off valve g.
- (vi) Close shut-off valve a.
- (vii) Close all the shut-off values  $\underline{k}^*$  at the outlets of the regulators of the supply bottles.
- (viii) If pressure of the gas being fed in is greater than 100
  psig, close shut-off valve e to protect low pressure
  test gauge #1.
  - (ix) If pressure of the gas being fed in is greater than 600 psig, detach the line <u>h</u> from the flow path and cap off the tee connector. This is for protection of gauge #2.
  - (x) Set the desired output pressure of the regulator on the supply gas bottle, say  $0_2$ , whose gas we now want to feed into D.

- (xi) Open the shut-off value  $\underline{k}$  downstream to the regulator mentioned in (x).
- (xii) Open the shut-off valve a.
- (xiii) Open the shut-off valve g.
  - (xiv) If gauge #1 or #2 is in service, read the internal pressure of <u>D</u> from them, otherwise, the high pressure gauge on regulator <u>b</u> has to be used for this purpose.
  - (xv) The pointer of gauge #1 (or #2) shows rising pressure when gas is flowing into bottle <u>D</u>. Equilibrium is achieved when no more upward movement of the pointer is detected.
  - (xvi) The reading on the test gauge #1 (or #2) should be used as fine adjustment for the desired partial pressure of the gas being introduced into bottle D.
- (xvii) Close shut-off valve g.
- (xviii) Close shut-off valve a.
  - (xix) Close shut-off valve k as reverse of step (x).
    - (xx) Repeat steps (viii) through (xix) for another gas with different (but always higher) output pressure than that which was last set in step (x).

\* Shut-off values k should be provided for isolating each regulator on the supply bottles to prevent possible damage of its output (low) pressure gauge caused by the back pressure from the output of other supply bottles.



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SCHEMATIC DRAWING OF THE **APPARATUS** 

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FIG. I



FIG. 2. MINIBEAM SYSTEM



FIG. 3 QUARTZ TUBE DISSOCIATED GAS SOURCE



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FIG. 4 : HOOK-UP OF THE MICROWAVE POWER SYSTEM





FIG 6







(a) COUPLED TO FORWARD POWER



(b) COUPLED TO REFLECTED POWER

FIG. 8 INSERTION OF DIRECTIONAL COUPLER TO MICROWAVE TRANSMISSION LINE



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RESULTS FROM DRY GAS SOURCE

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# TABLE I EXPERIMENTAL RESULTS

NOZZLE SIZE (1/1000)"	NOZZLE PRESSURE (TORR)	FLOW RATE (ATM.C.C. /sec)	NUMBER OF CAVITY	TOTAL INCIDENT POWER (WATT)	DEGREE OF NOZZLE COOLING	INCREASE IN 0/0 <sub>2</sub> RATIO	TEST Nº
	201	623	*	150	I	.0735	1
	20,1	.623		150	2	.0685	2
9					I	.0348	3
	47.75	1.062	I (C)	150	2	.0385	4
	83.8	1.861	I (C)	150	I	.0035	5
					2	.0055	6
15	25	2.575	I(C)	150	3	.078	7

NOTES (1) GAS MIXTURE WAS 7.89% 02 & 92.1% He BY VOLUME

(2) GAS MIXTURE WAS DRY IN TESTS 1-7

\* ONE CAVITY CENTRAL POSITION

NOZZLE SIZE (1/1000)"	NOZZLE PRESSURE (TORR)	FLOW RATE (ATM.C.C./sec)	NUMBER OF CAVITY	TOTAL INCIDENT POWER (WATT)	H20/02 RATIO	NUMBER OF TURNS AT H <sub>2</sub> O VAPOR CONTROL VALVE	DEGREE OF NOZZLE COOLING	INCREASE IN 0/0 <sub>2</sub> RATIO	TEST N≗		
	1999 (P. 1997)		I(S) *	N1591	10.0 mg		3	.0332	8		
			1(C)				- Loost	.0242	9		
	الو جادب		I(S)	135		and the second second	4	.0297	10		
			196				3	.0390	П		
	5	1.164	res alla		.099	3	4	.0415	12		
			2	140			3	,0576	13		
29.2				2	2	150		and the second		.1168	14
				150			4	.0873	15		
A Carton and				and the second			.1088	I <b>,</b> 5		.1520	16
	a sala				.1168	2		,1475	17		
	4	.931	2	150			4	.0937	18		
			and an	175	.1273	3		.0890	19		
CLUB CALL		a la statistica de la seconda de	2	150			20122-01-20	.1733	20		
	3.2	.745	1(C)	135	. 1578	1,5	4	.1463	21		

\* ONE CAVITY, HOOKED AT SIDE POSITION

Toronto
c Oxygen
14 figures
lation 3. Atomic Oxygen
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ng a hypersonic molecular beam to have been successfully completed. beular beam which could be used for reaction of the earth's upper atmos- prements or surface reactions at s developed for the molecular beam be maintained. The gas from the vas expanded into a free jet which nozzle beam techniques. The per- s measured using a mass spectrometer luded the effect of pressure and ori- i in order to achieve high Mach of double cavities, and the effect ad in these initial tests that a the produced with 17% dissociation. antially with further optimization.
Toronto
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. 153
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