NOTICE

PORTIONS OF THIS REPORT ARE ILLEGIBLE.
It has been reproduced from the best
available copy to permit the broadest
possible availability.

UCRL-15441
S/C 8678003

MASTER

Continued Investigations into Techniques Producing Selective Chemical
Reactions on Surfaces and Target Spheres and Related Studies

M. Bonner Denton
Department of Chemistry
University of Arizona

UCRL--15441

DE 2 012070

DISCLAIMER

This document contains information which is the property of the University of California, Lawrence Livermore Laboratory. It is loaned to you for your personal use only. It is not to be distributed outside your organization. It is not to be used for any purpose other than the one for which it was loaned to you. It is not to be reproduced, stored in a retrieval system, or transmitted in any form or by any means, electronic, mechanical, photocopying, recording, or by any information storage and retrieval system, without the prior written permission of the University of California, Lawrence Livermore Laboratory.

February 19, 1982

**Lawrence
Livermore
Laboratory**

NOTICE

PORTIONS OF THIS REPORT ARE ILLEGIBLE.
It has been reproduced from the best available
copy to permit the broadest possible availability.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

MGW

Final Report Subcontract 8678003

"Continued Investigations into Techniques Producing Selective Chemical
Reactions on Surfaces and Target Spheres and Related Studies"

Submitted to:

University of California
Lawrence Livermore National Laboratory
P.O. Box 5012
Livermore, California 94550

From:

M. Bonner Denton
Department of Chemistry
University of Arizona
Tucson, Arizona 85721

Sent to Wayne Johnson
LLL 5-7-80

Progress Report
Lawrence Livermore Laboratory

I. Introduction

One possible approach to the fabrication of inertial confinement fusion targets with high quality surface finish involves coating the glass microsphere while it is suspended in a quadrupole of rotation. Maximum flexibility in choice of coating composition, composition gradients, etc., should be possible if the coating materials are introduced in the form of reactive ion beams (1). Energy deposition in the target is also an important consideration and could be made optimal for an ion beam process. As presently envisioned, a high current electron impact source would generate several kinds of reactive organic ions, one or more of which can be selected for interaction with the target by means of a suitable mass analyser. Since high current densities of high mass ions must be focused on very small diameter target regions with low final beam energies, stringent requirements are placed on the beam handling and focusing optics.

This report describes efforts leading to the development and characterization of a compact ion source and optical transfer system producing relatively high current density ion beams. The ion source and beam transfer system represent a major advance in the state of the art in that high current densities at low kinetic energies have been achieved for high molecular weight polyatomic ions. Indeed, the ion beams produced

display ion abundance patterns typical of simple low energy electron impact ionization processes.

II. Overview

The development of the ion source and optical transfer system proceeded through several design generations. The first ion source geometry examined was a modified "Swingler" (2). Even with extensive design modifications, no more than a 4.5 microamps unfocused total ion yield could be produced with this source. Next, a source design based on a proposed electrostatic space propulsion unit (3) was built and characterized. In this case, more than a 12 microamps unfocused total ion yield could be produced. On the basis of this experience and extensive literature investigations, construction and characterization of a large volume electron impact ion source with an axial magnetic field to improve ionization efficiency was undertaken. Design concepts were drawn from both the NASA electrostatic propulsion unit and the Scandinavian or Penning ion source used in isotope separation and ion implantation equipment (4). In the third generation ion source the effects of magnetic cyclotroning on ion current yield were determined along with a first evaluation of the optical properties (focusability) of the ion beam produced. In the fourth generation design, the optical quality of the ion beam was improved and efficient focusing was achieved at final beam kinetic energies of 500 to 1000 eV. In the fifth and sixth generation ion source, design modifications were introduced to reduce gas residence times in the ion source region and allow differential pumping of the source region

with transverse gas feed. At the same time, an all-steel vacuum system was constructed which provided much cleaner ultimate vacuum and included a residual gas analysis capability via a General Electric monopole 300 mass analyser. The monopole also allows convenient evaluation of ion beam composition. Finally, in the sixth generation ion source variations in source geometry and filament composition led to the best performance observed to date. A deceleration lens was incorporated at this time to allow investigation of ion "soft landing" approaches to inertial confinement fusion target fabrication and examination of ion beam optical properties at very low kinetic energies.

Two approaches to inertial confinement fusion target fabrication have been attempted. Ion induced polymerization of neutral gases fed to the target region was examined in hopes of obtaining very high coating rates, but this approach was abandoned on receiving communication from Lawrence Livermore Laboratories that the technique was not of great interest. Ion soft lander results are preliminary at this writing, and samples of aluminum mirror targets coated with this procedure will be provided with the report. The sixth generation ion source with "soft lander" optics is able to produce focused current densities up to $70 \mu\text{A}/\text{cm}^2$ with a final ion kinetic energy of 50 eV. on impact. This should correspond to a film growth rate of about 1 micron per hour (see Appendix 1). Further refinement of the sixth generation source should lead to higher focused current densities. Work on beam mass analysis prior to deposition is currently under way.

11. Ion Source and Focusing Optics Development

The problems of ion source design have been the subject of intense effort by mass spectrometrists, accelerator physicists and those involved with semiconductor fabrication (ion implantation). The area has been the subject of several extensive reviews (5, 6, 7). Ion sources suitable for heavy ion accelerators and ion implantation equipment operate at high pressures and often high temperatures, producing an ionization environment in which extensive pyrolysis and/or chemical reaction often occurs (8, 10).

Conventional duoplasmatron and duopigatron guns produce high yields of atomic ions and small molecule ions, but will not produce substantial yield of polyatomic ions. In these sources, ions are created in a small volume region at relatively high pressures and extracted from the region with high draw out fields. This results in a high beam kinetic energy with a correspondingly large spread of ion kinetic energy (9, 10). RF discharge sources have similar problems (11, 12). The energy spread as a percentage of beam energy may be small, owing to the large acceleration voltages. The source emittance is also small, since ion current is drawn from a tiny aperture, typically 1 mm in diameter or less. Deceleration of the ion beam, however, degrades its properties to an intolerable degree. Some of these sources are, however, able to produce very intense ion beams, often on the order of milliamps. Focusable ion currents can be much less, however, particularly if the ultimate ion beam spot size desired is small and the beam kinetic energy is low. A small spot size

at low kinetic energy is extremely difficult to achieve. An important limitation is the energy spread in the ion beam produced by the ion source. Typical energy spreads are ~ 20 eV for the duoplasmatron and ~ 10 eV to ~ 50 eV for the hot and cold cathode Penning discharges, respectively (4, 13). The colutron ion source has been mentioned by Beynon as a possible candidate for analytical mass spectrometric application in those situations where high current is desirable (14). Unlike the duoplasmatron and Penning discharge ion sources, both of which employ magnetic plasma confinement or electron cyclotroning to achieve high plasma densities, the colutron source is simply a high current, low voltage arc discharge operated at relatively high pressures. Operating under these conditions, a considerable amount of pyrolysis and source chemistry occur. The colutron source is able to produce 0.11 eV energy spreads after electrostatic focusing to a 1.0 mm diameter spot, 40 cm from the source aperture (15). With a final kinetic energy of 200 eV, 1×10^{-6} A could be focused on a 1 mm diameter aperture. It should be noted that electrostatic focusing effects some reduction of energy spread downstream from the aperture as a result of lens chromatic aberration effects (16).

Electron impact ion sources for electrostatic space propulsion units and neutral beam injectors utilize electron cyclotroning in a weak magnetic field (1-10 gauss) to enhance ionization rates and generate a diffuse plasma which is extracted by a large area multielement lens (3, 17). The Scandinavian ion source operates in a similar fashion, but employs higher magnetic fields and extracts a conical beam from a small aperture (4).

Efforts directed toward more efficient, more intense ion sources for analytical mass spectrometers have generally involved quadrupole mass analysers which are much less sensitive to energy spread in the ion beam and to beam diameter than are magnetic sector instruments (18). General principles of source design for quadrupoles have been discussed by Brubaker (19) and Swingler (2), and the general field of electron impact, crossed beam ion sources has been reviewed by Valyi (20) and Wilson (21).

Given the above considerations, the sixth generation ion source reported here is designed around a low voltage hot cathode Penning discharge. This approach to source operation was selected after the previous investigations indicated that high intensity ion beams could be generated with acceptable energy spreads and good ionization efficiencies even at the low pressures (10^{-6} torr to 10^{-4} torr) desirable for true electron impact ionization with rather modest filament powers and magnetic fields. Indeed, a cold cathode Penning discharge, really a modified Redhead ion gauge, has been used successfully as an ion source for a quadrupole mass spectrometer, despite the high electron kinetic energies and intense magnetic field necessary for operation in the cold cathode discharge mode (22). The sixth generation source described later in this report can operate between 10^{-6} and 10^{-4} torr and, with careful design of electrostatic focusing optics, ion currents of up to 2 μ A have been focused through a 1.5 mm aperture with final beam energies of 500 volts. Most important, these high focusable ion currents are obtained under conditions compatible with the production of useful electron impact

spectra. Additionally, the observed energy spread is approximately 10 eV without prior electrostatic focusing and the form of kinetic energy distribution function demonstrates substantial ion current with a 0.1 eV range of the median beam kinetic energy, indicating potential application to magnetic sector instruments. This result may be surprising to those familiar with the Nier source design philosophy but is a well-known property of hot cathode "plasma" ion sources (20, 10).

Early in our ion source development work it was demonstrated that high ion current densities could be obtained only at high kinetic energies. To better understand the problem, let's have a brief look at what other investigators have been able to achieve to date, and then consider the factors limiting spot focused current density. Our approach to the problem will then be examined.

I am sure
this should
be (23)

The low energy, mass analysed ion beam deposition system reported by Yagi, et al (28) is capable of producing $100 \mu\text{A per cm}^2$ at 70 eV impact energy. This is the best low energy current density reported to date and with deposition of singly ionized germanium, a growth rate of 5.5 nm/min. is observed. Lawson reported substantially lower current densities for Pb^+ beams, on the order of $10 \mu\text{A/cm}^2$ at 70 eV with rapid deterioration of current density below 100 eV (29).

I am sure
this should
be 24

In both cases cited above, the investigators employed modified electromagnetic isotope separation equipment, and a total system cost on the order of $\$10^5$ to $\$10^6$ (30). Beam transport and analysis occur at 30 KeV, with mass analysis in a 60° magnet. It is significant that

the system reported here costs less than \$10⁴, produces a high yield of reactive polyatomic ions and is presently able to produce state of the art current densities in excess of 70 $\mu\text{A}/\text{cm}^2$ at 50 eV. Additional improvement in source performance is possible and work is under way in this area.

In an ideal optical system, the only limitation on the spot size produced by focusing an ion beam is space charge, and even this becomes negligible at sufficiently high beam kinetic energy. In any real ion optical system, however, aberrations in the beam focusing and analysis components combined with beam imperfections such as spread in ion kinetic energy result in a much larger minimum spot size than that expected for the ideal case. In general, the beam spot diameter can be expressed as a sum of independent random contributions. Thus,

$$\delta_T = (\delta_{s.c.}^2 + \delta_{sph.}^2 + \delta_{ch.}^2)^{1/2} \quad (21),$$

where δ_T is the best obtainable spot size for the system under study and $\delta_{s.c.}$ = contribution from space charge effects. $\delta_{sph.}$ = contribution from spherical aberration, $\delta_{ch.}$ = contribution from chromatic aberration.

It can be shown both theoretically (16) and experimentally (Figure 26) that space charge is negligible in the sixth generation system described below, while spherical aberration and chromatic aberration

are dominant factors. As shown in Figure 26, increasing the beam kinetic energy in the final stage of the secondary focusing triplet invariably increases the current density focusable on a small diameter aperture. This is easy to understand in terms of the spherical aberration coefficient and the magnification factor for a triplet lens and how these quantities vary with lens acceleration factor v_3/v_1 . Harting and Reed (26) have calculated aberration coefficients and figures of merit for a variety of three element circular aperture lenses. The calculations show that the spherical aberration coefficient goes down while the magnification factor goes up, resulting in higher overall current density on a small aperture, i.e. acceleration with focusing always gives a smaller spot, as implied by Figure 26. An approximate calculation of the figure of merit for a decelerating lens shows that for convergence half angles less than 10 degrees and a deceleration factor of about 30, the high current density spot produced by the first accelerating triplet could be reimaged at $1/30$ the initial kinetic energy with negligible increase in spot size from spherical aberration. At low kinetic energies, however, chromatic aberration becomes important. Fortunately, the effect seems unimportant for final energies above 50 eV, as will be shown. Ion current does increase, however, with increasing final energy, probably due to a steady increase in the figure of merit for the lens, related, of course, to a steadily improving spherical aberration coefficient.

The First Generation Ion Source

Initial experimental studies involved characterization of the "Swingler" type ion source (2, 27, 28). This "high current" ion source was designed for use with quadrupole mass filter gas analysis and is capable of producing sensitivities of 12 amps per torr up to 10^{-4} torr.

Materials and Methods

The ion source was constructed completely from Kimbal Physics eVTM parts. Metal aperture plates, repeller structures and spacers were of type 304 stainless steel (electropolished), while insulators were made of Al_2O_3 . Emissive filaments were made of .010" Mackey tungsten wire and were spot welded to tantalum support posts. The ion source was mounted in an aluminum support frame. The support frame and ion source were placed in a Corning glass 6" pipe cross which was pumped by a Varian VHS6 backed by a Sargent Welch duo-seal model 1397 rotary vane rough pump. The vacuum flanges were made of aluminum and provided with electrical feed throughs. Technical grade fluorocarbon gases were obtained from Pfaltz and Bauer and were used without prior purification. System pressure was measured with a Veeco Ion gauge calibrated with N_2 and placed just above the gate valve leading to the glass vacuum chamber.

Apparatus Layout and Experimental Procedure

The ion optical and electrical layout of the Swinger ion source is shown in Figure 1. Ion current was measured with a 536T based FET op amp electrometer amplifier, with target bias relative to earth ground established with a dry cell battery. Total ion current received by the faraday cup was optimized by variation of lens element potentials in the drawout, focusing einzel lens facing the repeller volume. No fine focusing was attempted so the ion current values were for unfocused ion current.

Results and Discussion

Total unfocused ion current is plotted vs. system pressure as measured with the ion gauge in Figure 2. While the design is certainly of high efficiency compared with more conventional sources, it cannot be operated at the levels required by this project. Only 4.3×10^{-6} amps of unfocused ion current could be produced under optimum conditions. (Subsequent ion sources described in this report are able to focus 3.35×10^{-6} amps through a 1.5 mm diameter aperture.) The Swinger ion source was therefore abandoned, though experience gained in source operation and construction proved valuable in subsequent studies.

Figure 1. Ion Source configuraticn, The Swinger Ion Source. Operating conditions for ion current data shown in Figure 1.
 Filament Current - 6.0 Amps at 16 volts/Filament bias -50.volts
 Emission Current - 15 milliams
 Source Voltages: 1) Repeller - +124v, 2) Ion cage - 0.0.volts, 3) Ion Extraction Electrode/
 Ion exit aperture - -170 volts

Figure 2. Positive ion current versus ion gauge indicated system pressure in torr. For operating conditions see the figure one legend.

The Second Generation Ion Source

The next developmental trial structure was based on concepts proposed for a space vehicle propulsion unit (3). A cutaway sketch of the propulsion unit that inspired the second generation design is shown in Figure 3. Some important differences exist between the two designs, and these will be detailed in the apparatus section below. The design was motivated by the high ion currents available from the ion rocket, relatively low pressure large volume ionization environment compatible with true electron impact ionization and a cylindrical geometry compatible with electrostatic spot focusing. In this first model of the series to follow, magnetic cyclotroning was not attempted, but even so, much higher unfocused ion currents than the Swingler source produced were available. An important feature of this source geometry is the fact that electron cyclotroning with an axial magnetic field is easily implemented.

Materials and Methods

The vacuum system used was the same as that previously described for the first generation ion source. The ion source structure and faraday cup assembly were constructed entirely of "homemade" components. The anode cylinder and extraction-focusing optics were made of cut lengths of stainless steel pipe. The gas feed structure at the rear of the anode cylinder was made of aluminum with the 36 gas delivery holes in a circular pattern .82" radius. The holes were made with a #59 drill. The entire ion source assembly and the faraday cup were mounted on an aluminum "optical

Figure 3. The Ion Propulsion Unit

rail" with ceramic isolation posts having blind holes for 6-32 screws.

The ion source feed gases were the same as those used in operation of the first generation ion source.

And not AUT

Apparatus Layout and Experimental Technique

The apparatus layout is shown in Figure 4. The electrical layout is the same as that used with the first generation source. The unipotential drawout and focusing lens was retained in this design. While some focusing was necessary to optimize the ion current into the faraday cup, the large aperture of the faraday cup (1.5") once again results in a total ion current measurement, not a focused optimized current density measurement. Gas pressures were measured, once again, with a Veeco ion gauge calibrated with N_2 .

Results and Discussion

Ion current vs. pressure data are shown in Figure 5. Comparison of this data with corresponding results obtained with the first generation source demonstrates a much higher total ion yield with the second generation geometry than could be obtained with the first 12 microamps as opposed to 4.3. The next step, inclusion of magnetic cyclotroning of the electrons in a true oscillating electron geometry, was taken in the third generation VHY source described below.

Figure 4. The Second Generation Ion Source. Experimental layout.

Figure 5. Positive ion current versus ion gauge indicated system pressure in torr. For operating conditions see the figure three legend.

The Third Generation Ion Source

The third generation ion source incorporates several fundamental changes in design concept. Electrons are introduced into the ionization volume, i.e. the anode cylinder, from an emissive filament mounted to the rear of the anode cylinder. An anticathode, held at or below filament potential, reflects electrons back into the anode cylinder volume where they spiral out toward the anode under the influence of an axial magnetic field. The anticathode is also employed as the first stage in ion extraction optical system. Two subsequent lens elements prepare the ion beam for ultimate fine focusing with a secondary focusing triplet used to optimize ion current through a small axial aperture and so maximize ion current density at some selected final kinetic energy.

These concepts somewhat superficially resemble a "hot cathode Penning" or "oscillating electron" discharge. Several major design differences exist between the classical Penning ion source and Very High Yield (VHY) source described below. These differences include:

- 3
- (1) The cathodic mirror arrangement is unsymmetrical, i.e. $V_{c1} \neq V_{c2}$. The ability to independently set the filament potential and anticathode potential were found to be critical in optimization of focused current densities in subsequent ion source generations. (V_{c1} is the potential relative to reference ground of the filament mount plate and V_{c2} the potential relative to ground of lens A of the unipotential section, which

would correspond to the anticathode in a Penning gun.)

- (2) No special gas feed arrangements are made in this source. It is operated in what is essentially an "open source" configuration. An improved gas feed arrangement like that discussed for the second generation ion source report (2) is introduced for future models, but proved to be nonessential for source operation.
- (3) Relatively low magnetic fields are employed to cyclotron the electrons and no effort is made to focus magnetic force lines near the extraction aperture by employing a soft iron lens. Improved magnetic field geometries will be examined in the future.
- (4) The ion extraction-focusing arrangement used here allows high drawout fields and high ion extraction efficiency without necessitating high ultimate ion kinetic energy.

It should be noted that this ion source provides unique capabilities in the combinations of a high useable ion beam yield at a low kinetic energy.

Materials and Methods

The vacuum system used in this study consisted of a 4" CVC oil diffusion pump using Corning 704 silicone oil as the working fluid

backed by a Kenney KC-8 roughing pump. Two cryobaffles were included in the system: 1) a Chevron cryobaffle between the diffusion pump and the gate valve leading to the vacuum chamber, and 2) a foreline trap between the KC-8 and the oil diffusion pump. A Corning glass, pyrex 6" pipe tee was used as the vacuum chamber. The ion source chamber a water-cooled stainless steel vacuum nipple, $2\frac{1}{2}$ " o.d., $2\frac{1}{8}$ " i.d., and $4\frac{3}{8}$ " long with homemade brass flanges on each end was bolted to an 11" diameter aluminum flange via blind bolt holes. The 1" thick aluminum flange was countersunk on the vacuum side so that the thickness was $\frac{1}{2}$ " for a 5" diameter, and provided with an eVTM parts C7X7 hole pattern of blind tapped 4/40 thread holes for attaching ion source structures. The water-cooled nipple bronze end flange had a similar 4/40 thread hole pattern for C5X5 parts also for ion source mounting. The filament was made of $8\frac{1}{8}$ " turns of .010" Tungsten (Mackey). The anode cylinder is positioned with four alumina rods (eVTM part Al₂O₃-Tu-C-4000) and alumina or glass ceramic insulators (eVTM Al₂O₃ or C1L-Sp-C-175). The insulators also serve to space the elements of the unipotential extractor lens assembly, as shown in Figure 6.

Since lensing potentials were brought from the vacuum side of the large aluminum flange, a grounded shield was necessary to prevent ion beam distortion and deflection. A stainless steel cylinder (eVTM part no. SS-Cy-1250/2500) is suspended from a mounting plate (eVTM part no. SS-PL-CyXy-R1250) so that the cylinder orifice was $7/16$ " from the last element of the extractor unipotential lens. Four blind 4-40 tapped holes on a $2\frac{7}{16}$ " bolt circle concentric with the center hole in the

aluminum flange were used to fix a mounting plate with 4-40 S.S. screws and three eV^{TM} Al_2O_3 or GL-Sp-C-175 insulators. Four eV^{TM} parts alumina rods (part # Al_2O_3 -Tu-C-3000) were used to align the beam shield, retarding grid and faraday cup with part no. GL-SP-C-175 insulators used to space these components, as shown in Figure 6.

Apparatus Layout and Experimental Procedure

The apparatus layout used in total ion current measurements and ion kinetic energy distribution measurements on the total "unfocused" output of the third generation VHY ion source is shown in Figure 6. Ion kinetic energy distributions were determined by the retarding field method. As increasingly positive potentials were placed on the retarding grid, the fractional decrease in ion current reaching the faraday cup was recorded. Given the large aperture of the faraday cup, 1.5", the ion currents reported represent total ion source output, not best focus values. The kinetic energy spread is also representative of total source output with no kinetic energy filtering characteristic of electrostatic fine focusing.

The apparatus layout used in fine focusing studies is shown in Figure 7. The deflector plates proved necessary since optimal axial alignment proved difficult with this source mounting geometry and optical train assembly,

Ion currents were measured with a 536T based FET input electrometer operational amplifier with target bias established using dry cell batteries.

Figure 6 - Layout of the ion gun, beam line, and detector. Dimensions and eV^{TM} part numbers are given in the text. Vacuum system detail has been omitted, along with solenoid positions for clarity. The anode cylinder extends $1\frac{1}{2}$ " from behind into the solenoid which is itself $2\frac{1}{2}$ " long.

Figure 7. Drawing of "Third generation" VHV ion source, lenses, deflection plates, mask, and target.

Figure 7A. Photograph of Third Generation ion source, deflection plates, etc. (Silicon substrate collector has been removed.)

3

The instrument layout used for crude mass spectra using a home-built quadrupole mass spectrometer described elsewhere (28) is shown in Figure 8. Mass spectra were recorded with a simple analog linear mass scan over the 0 to 120 amu mass range.

Ion source assembly is described in some detail below. Except for the cyclotroning magnet, the source can be constructed completely from Kimball Physics eVTM parts. The most important advances involve addition of electron cyclotroning and improvements in source geometry. The most important improvements in source geometry have been made in the filament mounting and the relative placement of filament and anode. An eVTM SS-PL-C5X5-S1000 mounting plate was attached to the brass base plate via blind 4-40 tapped holes on the vacuum side, the plate being held 7/16" off the surface with ceramic spacers as shown. The 4-40 hole circle is concentric with gas feed port. Electrical feed throughs were achieved with Kings "vacuum" BNC connectors.

The top of the filament mounting posts is positioned 1/4" behind the anode cylinder as shown in Figure 5. The filament mounting assembly is spot welded to an eVTM SS-PL-C5X5-R750 plate as shown. The filament mounting plate is electrically connected to the negative filament terminal.

Technical grade fluorocarbon gases, octafluoro-2-butene, decafluorobutane, and hexafluoroethane were obtained from Pfaltz and Bauer and used without prior purification. Mass spectral analysis showed the gases to be better than 95% pure.

Figure 8. Apparatus layout for Mass Spectrometry of Third Generation VUV Ion Source Output

Results and Discussion

Source performance data were collected under a variety of different operating regimens. Ion current at several different system pressures, with no cyclotroning magnetic field present, is shown in Figure 9. Experimental conditions are given in the figure captions. A decrease in ion current was observed with both fluorocarbon gases used whenever the system pressure exceeds 5×10^{-4} torr. Emission current, i.e. electron current collected at the anode cylinder, was set at 10 milliamps at 2×10^{-5} torr and was observed to go off scale at a pressure of 2×10^{-4} torr of C_4F_8 . With octafluoro-2-butene the sharp increase in emission current indicates a sudden increase in secondary electron yield, i.e. formation of a discharge. Increasing the negative bias on the faraday cup from -90 volts to -156 volts resulted in some increase in indicated positive ion current, suggesting that the ion beam contains substantial number of electrons. Increasing filament diameter and wattage for higher emission currents coupled with one of several possible techniques for removal of electrons from the ion beam should make ion currents as high as 100 microamps easily achievable with no magnetic cyclotroning, although higher system pressures would be required.

Application of the cyclotroning solenoidal magnetic field to the thermionic emission discharge described above resulted in considerably more complex source behavior. Filament current, solenoid current, extraction field and gas pressure all strongly influence ion current and all have sharply defined optimum values. At very low pressures,

Figure 9 - Ion current v.s. system pressure for two Fluorocarbon gases. O - octafluoro-2-butene. O· - hexafluorothane. Pressure was determined with a Bayard-Alpert ion gauge calibrated for H_2 .

Operating conditions: Filament bias is -165V, Anode bias is +135V, lens A,C held at -1KV, lens B at 0V, faraday cup bias -90 volts, filament current 5.5 amps.

less than 5×10^{-5} torr, an increase in the solenoid current results in a steady increase in positive ion current up to approximately 150 gauss. As the solenoid current was increased further, positive ion current decreased to zero and finally electron current was observed. Ion current vs. magnetic field data were taken at several different pressures and, as shown in Figure 10, the magnetic field corresponding to optimum ion current is lower at high pressures. Above 3×10^{-4} torr, ion current steadily decreases from an optimum value as magnetic field is increased. At B fields in excess of 500 gauss a penning plasma may form between the beamline and faraday cup. Increasing the drawout field voltage to the present available maximum of -2Kv, suppressed formation of the discharge and with the magnetic field present allowed production of 50 microamps of positive ion current at 1×10^{-4} torr of octafluoro-2-butene. It should be noted that drawout field is defined as the potential on lens A, C in Figure 6. Since these lens elements are a portion of an unipotential lens they do not accelerate the ion beam. Beam kinetic energy is defined by the potential applied to the anode, probably by changing ion origin conditions through changes in plasma density and distribution in the cylinder region.

The ion kinetic energy distribution and focusing data reported below both support the notion that the ion source region is a "plasma" as in higher pressure hot cathode penning ion sources. Indeed, the formation of a plasma in the source region is probably responsible for narrow kinetic energy spread compared to that expected

Figure 10 - Ion current v.s. magnetic field at three different pressures. ▲ - 5×10^{-5} torr, ○ - 2×10^{-4} torr, ○ - 5×10^{-4} torr.
Operating conditions: Filament bias is -165V, Anode bias is +135 volts, lens A,C held at -1KV with lens D at 0V, faraday cup bias, -90 volts, filament current 5.6 amps.

for an "Ion Cloud" source of similar geometry (21). It should be noted that increasing the emission current without magnetic field does not result in a large increase in focusable ion current in this device.

Ion kinetic energy distribution was determined by the retarding grid method, and results are shown in Figure 11. The full width at half maximum is less than 15 eV with an average ion kinetic energy of approximately 30 eV relative to reference ground.

The kinetic energy spread would seem to imply that high focused current densities might be obtained. With 1×10^{-4} torr of octafluoro-2-butene, 0.14 microamps could be focused through a 1.4 mm diameter aperture with a final beam kinetic energy of about 70 eV for a focused current density of 9.8 microamps per cm^2 . (This value will be improved by nearly an order of magnitude in subsequent VHY ion source systems.)

Lastly, the ion beam composition was determined with a homebuilt quadrupole mass spectrometer in the 0 to 120 amu mass range and fragmentation patterns produced by the third generation ion source compared with those produced by the Swinger type first generation ion source. Results are shown in Figures 12 through 15. Mass spectra of octafluoro-2-butene and hexafluoroethane are shown. The third generation VHY source does exhibit slightly enhanced fragmentation relative to the Swinger, but this is expected as a result of different electron kinetic energies in each source. In the Swinger, electron impact kinetic energies vary continuously between filament-repeller potential and zero

Figure 11 - Ion kinetic energy distribution as determined by a retarding field method.

Operating conditions.

Filament current : 5.2 amps.
 Filament bias : -117 volts.
 Anode bias : +88 volts.
 Magnet current : .75 amps.
 Magnetic field : 165 gauss.
 Faraday cup bias : -85 volts.
 Gas, pressure : Octafluoro-2-butene, 1×10^{-4} torr

Figure 12 Substance source spectra of hexafluorothene.

Figure 13 Mass spectrum of hexafluorobenzene.

Figure 14 Plasma gun mass spectra of octafluoro-2-butene.

Figure 15. Gas-liquid chromatogram of octafluoro-2-butene.

electronvolts. In the VHY source, on the other hand, electrons undergo nearly full anode-cathode potential in the cathode sheath, a region only a fraction of a millimeter thick (21) since a plasma is formed in the source ionization volume.

The most encouraging observation was that the major improvement in ion yield given by the magnetic gun resulted in a real improvement in throughput. Electrometer gain could be reduced three orders of magnitude relative to the gain necessary when using the Swinger. This was achieved despite poor alignment and without differential pumping.

In conclusion, the third generation ion source has demonstrated the essential viability of the low pressure oscillating electron source design concept. Subsequent VHY ion source design generations discussed in the following demonstrate a steady progress in all source performance areas.

]

The Fourth Generation Ion Source

Several design modifications suggested by experience with the third generation source as well as additional theoretical considerations based on lens properties calculated using the techniques presented by Harting and Reed (26) were incorporated in the fourth generation source. A larger water cooled vacuum housing was built to allow use of a C 7X7 series eVTM parts. Using larger parts means larger apertures in the electrostatic optical train as well as ionization in a larger volume (with a 1.5 inch O.D. anode cylinder). This size scale was employed in all further ion source development work. Another important advantage of the new larger source chamber arises from the fact that the entire optical train and ion source could be built on the same flange, providing improved optical alignment so that the deflector plates used in the third generation source could be dispensed with. This source was used for more detailed examination of the kinetic energy distribution as a function of anode-cathode bias, as well as focusing studies. In addition, some film formation work with the ion induced polymerization procedure was done using this source and beam line.

Materials and Methods

The internal components of the ion source and all optical train elements were made from Kimbal Physics eVTM parts. Metal components were type 304 stainless steel with 1/4" copper tubing silver soldered to the exterior

surface to provide the required water cooling. The ion source housing was mounted on an 11" diameter aluminum vacuum flange, allowing the entire assembly to be mounted on a Corning glass (pyrex) 6" x 6" glass pipe cross. The cross was mounted on a vacuum pumping station equipped with a CVC 4" oil diffusion pump backed by a Kinney KC-8 roughing pump. A Chevron cryobaffle was located between the oil diffusion pump and the gate valve leading to the source chamber. Another cryotrap was placed between the roughing pump and the diffusion pump. Pressure measurements were made with a Veeco RG ion gauge calibrated with N_2 and mounted on the 6" glass cross via a compression fitting feedthrough on a side arm flange made of brass. The solenoid magnet was homemade, consisting of a 5.5" I.D. aluminum cylinder wrapped with #14 AWG motor wire insulated with heavy duty laquer. The windings were held in place by 1/16" thick mild steel plates forming a 3" wall on each end of the aluminum cylinder. The magnet was wound on an engine lathe in back gear until a layer of windings 3" deep had been applied. The axial field at several different current settings was determined with a calibrated hall effect probe. Decafluorobutane, hexafluoroethane, and octafluoro-2-butene were obtained from Pfaltz and Bauer, Inc. This batch of technical grade gases was also analysed by mass spectrometry and found to be more than 96% pure. Specific experimental arrangements are discussed below.

3

]

Apparatus Layout and Experimental Procedure

Apparatus layout for ion kinetic energy and total ion yield studied is shown in Figure 16.. Ion kinetic energy distributions were determined by the retarding field method. A high transparency tungsten woven cloth grid was placed in the beam path to the faraday cup and total ion current was recorded as the positive bias voltage on the grid was varied. The cathode structure was held at -150 volts while the anode structure potential was varied between +103 and +13.4 volts. All voltages are measured relative to local earth ground. The system pressure was maintained at 2×10^{-4} torr for all studies.

Focusing studies were conducted with the apparatus shown in Figure 17. Lens spacings and potentials in the secondary triplet were selected to approximate values suggested by Harting and Reed (26) for 3 element circular aperture lenses. The motives for selection of these particular spacings were two: 1) to allow calculation of lens potentials for best focus and allow sizing of lens power supplies, and 2) to compare best focus values with calculated values to see how useful the Harting and Reed calculations would ultimately be in future design work, given the poor "ideality" of any real ion optical system.

The apparatus layout used for ion beam induced polymerization studies is shown in Figure 18. Typically, a first surface AL mirror provided by Lawrence Livermore Laboratory and cleaned with the recommended procedure was placed in the target chamber in place of the faraday cup with a neutral gas feed provided by a 1/16" stainless tube adjacent to the target

Figure 16. Fourth Generation VHY Ion Source. Experimental layout for ion kinetic energy distribution measurements.

Figure 17. Fourth Generation VHY Ion Source. Apparatus layout for focusing studies.

Figure 18. Fourth Generation VHY Ion Source. Apparatus layout for Ion Beam Induced Polymerization.

and isolated from the target electrically with alumina spacers.

Results and Discussion

The ion kinetic energy distributions at the several different anode potentials are shown in Figure 19. The data show that increasing anode-cathode bias in the manner described results in an increasing energy spread and a higher median beam kinetic energy as shown in Figures 20 and 21. It should be noted that increasing anode potential and hence anode-cathode bias leads to an increase in the magnetic field required for optimum ion current. With the entire gun structure referenced at ground potential, the median kinetic energy is found to be $0.8 \times$ (anode cylinder potential). All these results are consistent with previous observations of the penning discharge (21).

Further, the data indicate that a substantial improvement in ion current is possible for both sector and quadrupole mass analysers in comparison to that available from conventional electron impact ion sources, resulting in higher focusable ion current after mass selection. However, kinetic energy spread is not the only factor limiting beam quality. Focusing experiments were conducted with the apparatus shown in Figure 17 to better define beam quality. Ion currents of 3.5×10^{-6} amps could be focused through a 1.5 mm diameter aperture with a system pressure of 2.5×10^{-4} torr hexafluoroethane and a beam kinetic energy of 1.0 keV at the target aperture. At a final kinetic energy of 500 eV only 0.8×10^{-6} amps of ion current could be focused through the aperture, while at

Figure 19. Ion kinetic energy distribution at several anode potentials as determined by the retarding field method.

Figure 20. Ion beam median kinetic energy as a function of anode cylinder voltage.

Figure 21. Full width at half maximum of the ion kinetic energy distribution vs. anode cylinder potential.

700 eV and 460 eV only 1.0×10^{-6} amps and 0.50×10^{-6} amps, respectively, could be obtained. 15×10^{-6} amps total ion current was produced under these conditions, so between 10% and 20% of total source output could be directed through the aperture for a current density of 245×10^{-6} per cm^2 with final beam energy of 1.0 KeV.

Since chromatic aberration due to energy spread can be shown to be negligible under these conditions (26), spherical aberration is the most likely cause for the rapid decay in current density with final beam kinetic energy. A brief perusal of lens figures of merit in reference 26 demonstrates that spherical aberration increases rapidly with decreasing final kinetic energy of the beam, especially where large magnification factors are involved. Data taken with subsequent ion source design generations support this conclusion.

Coating studies were initiated with this source in an ion beam induced polymerization approach following observation of "thick" films on steel target chamber surfaces. Results are summarized in Table I. Lack of residual gas analysis made detailed analysis difficult, but the fact that no films formed under cryopumped conditions suggests a role played by low volatility species formed in the gun plasma or perhaps residual pump oil. Surface analysis of films formed in this manner indicated a C_1F_1 stoichiometry and a very high electric resistance, greater than metal oxides of similar film thickness. These facts, coupled with the importance of neutral feed in ultimate film thickness, tend to support a role of plasma polymerization in the gun region and discount the direct contribution of pump oil to film formation, a

Coat Designation (Notebook Pages)	Gun Gas (Base Pressure) Producer and Lot No. Partial Pressure	Ion Current; Ion Kinetic Energy at Target	Electron Kinetic Energy in Gun Plasma	Extractor Voltage; Anode Voltage; Decel Voltage	Neutral Gas Producer and Lot No. Partial Pressure	Focusing Conditions A, B, C. Sec. Trip.	Target Preparation in Vacuum Chamber	Results
SLK 87 4th Gen. Gun	Octafluoro-2-butene Pfaltz & Bauer Lot # 1 x 10 ⁻⁴ torr.	15 μ A 0 ev.	200 ev.	-500 0 -500		-24, +175, ond.	Silicon Target	4 $\frac{1}{2}$ hrs. Film < .5 μ Comp. unknown.
SLK 91 4th Gen. Gun	Octafluoro-2-butene Pfaltz & Bauer Lot # 2 x 10 ⁻⁴ torr.	25 μ A 0 ev.	200 ev.	-240 0 -240		-24, +360, ond.	Silicon Target	4 $\frac{1}{2}$ hrs. Film < .5 μ
SLK 128 6th Gen. Gun 3-1-79	1 butene (2 x 10 ⁻⁵) Lot #883 Filler F.C. Lot # 1.6 x 10 ⁻⁴ torr.	3.50 μ A 150 ev.	400 ev.	-2 KeV. -6.9 volts -1.56 KeV	1.4 x 10 ⁻⁴ 1-butene; same as gun gas	-14, +20, -600	None	5 hrs. Film produced but poorly adherent to target. < 1 μ thick
SLK 130 6th Gen. Gun 3-2-79	Octafluoro-2-butene Pfaltz & Bauer Lot #000646							1 hr. only--Film visible on target--removable by isoprop & rubbing
SLK 131 6th Gen. Gun 3-5-79	No coat run or ion current recorded--Gave up voltage divider geom. Started using August Johnson's $\pm$ 500 volt. biasing supplies.							
SLK 133 6th Gen. Gun 3-7-79	No cryopumping. Gun failure after 1 hour due to carbon on insulators 2-5 μ A last current.							
SLK 134 6th Gen. Gun 3-9-79	Hexafluoroethane from Matheson (1 x 10 ⁻⁵) Lot # 1.5 x 10 ⁻⁴ torr. Lot #98	1.37 μ A 166.6 ev.	540. 6 ev.	-2 KV. +10.6 ev. -2 KV.	.5 x 10 ⁻⁴ torr. Same as gun gas. Side mount geom.	-5, +87, -500	None	6 hrs. Adherent film. 50% C. by analytical ctr. Prob. < 1 μ thick
SLK 135 6th Gen. Gun 3-11-79	Hexafluoroethane from Matheson Lot # 1.5 x 10 ⁻⁴ torr. Lot #98	1.40 μ A 168.8 ev.	380 ev.	-2 KV. +29.8 V. -2 KV.	.5 x 10 ⁻⁴ torr. Same as gun gas. Jet-top geom.	-6.6, +177, -500	None	6 hrs. Adherent film thinner than 134 on side closest to neutral jet
SLK 139 6th Gen. Gun 3-15-79	Hexafluoroethane from Matheson (1 x 10 ⁻⁵) Lot # 1.5 x 10 ⁻⁴ torr.	.94 μ A 487 ev.	721 ev.	-2 KV. +336 V. -700 V.	1.5 x 10 ⁻⁴ torr. Same as gun gas. Side mount geom.	+1.9, +183, -500	None	6 hrs. "Thick" adherent film like SLK 134. Sent to NRA for Auger Spect.
SLK 141 6th Gen. Gun 3-25-79	Decafluorobutane Pfaltz & Bauer Lot # 1 x 10 ⁻⁴ torr.	.538 μ A 646 ev.	915 ev.	-1 KeV. +495 V. -1.9 KeV.	.5 x 10 ⁻⁴ torr. Same as gun gas.	-40, +101, -500	None	6 hrs. Film about .5 μ by Univ. SEMQ center. C.F. approx. stoik. 1.1

Table I. Coating studies employing the fourth generation ion source.

Coat Designation (Notebook Pages)	Gun Gas (Base Pressure) Producer and Lot No. Partial Pressure	Ion Current; Ion Kinetic Energy at Target	Electron Kinetic Energy in Gun Plasma	Extractor Voltage; Anode Voltage; Decel Voltage	Neutral Gas Producer and Lot No. Partial Pressure	Focusing Conditions A, B, C. Sec. Trip.	Target Preparation in Vacuum Chamber	Results
SLK 143 6th Gen. Gun. 3-30-79	Decafluorobutane <2 x 10 ⁻⁵ Pfaltz & Bauer Lot # 1 x 10 ⁻⁵ torr.	1.5 μ A 554 ev.	796 ev.	-1298 V. +398 V. -930 V.	.5 x 10 ⁻⁵ torr. Octafluoro-2-butene Pfaltz & Bauer	-40, +101, -500	None	6 hr. Film poorly adherent & seems resputtered near ctr. No analysis.
SLK 145 6th Gen. Gun 4-2-79	Decafluorobutane <2 x 10 ⁻⁵ Pfaltz & Bauer Lot # 1 x 10 ⁻⁵ torr.	.7 μ A 548 ev.	796 ev.	-1298 V. +398 V. -930 V.	.5 x 10 ⁻⁵ torr. Octafluoro-2-butene Pfaltz & Bauer	-40, +101, -500	30 min w/o neutral feed gas	6 hr. Relatively thick film by SEMQ = 2.5 μ at ctr. C ₁ F ₁ less than
SLK 146 5-3-79	Decafluorobutane <2 x 10 ⁻⁵ Pfaltz & Bauer Lot # 1 x 10 ⁻⁵ torr.	.7 μ A 548 ev.	796 ev.	-1 KeV. +398 -580	.5 x 10 ⁻⁵ torr. Octafluoro-2-butene Pfaltz & Bauer	0, +100, -500	30 min w/o neutral feed gas	5 hr. Like SLK 145 by SEMQ = 2 μ at center.
SLK 157 6-6-79 Long duration run for LLL shipment	Hexafluoroethane <2 x 10 ⁻⁵ from Matheson Lot #98 1.9 x 10 ⁻⁵ torr.	206 ev. 2.2 μ A 6 hrs. Replace filament. 206 ev. 2.0 μ A 8 hrs. 356 ev. 2.5 μ A 8 hrs.	550 ev.	-1020 +50 -1900	1.1 x 10 ⁻⁵ torr. Octafluoro-2-butene Pfaltz & Bauer	-15, +55, -500	15 min of Art at .2 μ A I _{ke} = 596 ev. Then 10 mn at 4 μ A w/ 1000 ev = 1 st ke.	Film > 4 μ by SEMQ v 7 μ center 23 hours
SLK 159 7-2-79	Hexafluoroethane <2 x 10 ⁻⁵ from Matheson Lot #98 1.9 x 10 ⁻⁵ torr.	3.5 μ A 350 ev.	227 ev.	-1230 +102 -700	1 x 10 ⁻⁵ torr. Octafluoro-2-butene Pfaltz & Bauer	-5, +117, -640	15 min of Art at .2 μ A I _{ke} = 596 ev. Then 10 mn at 4 μ A w/ 1000 ev = 1 st ke.	2.9 μ by interference at LLL. 30 hours in 3 10-hr blocks.
SLK 161 7-16-79 7-16-79	Hexafluoroethane <2 x 10 ⁻⁵ from Matheson Lot #98 1.9 x 10 ⁻⁵ torr.	.7 μ A 520 ev.	60 ev.	-854 +361 -1240	1.5 x 10 ⁻⁵ torr. Octafluoro-2-butene Pfaltz & Bauer	-50, +240, -600	1.5 μ A for 30 min at 1 KV	Film looked "thin" on removal from val sys. No anal. at LLL. All up on arrival.
SLK 163 -22-79	Decafluorobutane 1.5 x 10 ⁻⁵ torr. Pfaltz & Bauer (1.5 x 10 ⁻⁵)	.7 μ A 556 ev.	800 ev.	-750 +400 -1350	Octafluoro-2-butene .7 x 10 ⁻⁵ torr. Pfaltz & Bauer	0, + , -600	2 μ A Argon 1 KeV for 1 hour	20 hr. Film peeled up, apparently as formed when thickness > 2 μ

Table I (continued)

Coat Designation (Notebook Pages)	Gun Gas (Base Pressure) Producer and Lot No. Partial Pressure	Ion Current: Ion Kinetic Energy at Target	Electron Kinetic Energy in Gun Plasma	Extractor Voltage: Anode Voltage; Decel Voltage	Neutral Gas Producer and Lot No. Partial Pressure	Focusing Conditions A, B, C. Sec. Trip.	Target Preparation in Vacuum Chamber	Results
SLK 167 B-4-79	Decafluorobutane 1.5×10^{-4} torr. Pfaltz & Bauer (1.5×10^{-5})	.7 μ A 551.6	780 ev.	-704 +400 -900	Octafluoro-2-butene $.5 \times 10^{-4}$ torr. Pfaltz & Bauer	-45, +29, -710	2 μ A Argon 1 KeV for 1 hour	5 hr. Film peeled up, apparently as formed when thickness > 2 μ
SLK 169 B-5-79	Decafluorobutane 1.5×10^{-4} torr. Pfaltz & Bauer (1.5×10^{-5})	.7 μ A 551.6	780 ev.	-704 +400 -900	.5 $\times 10^{-4}$ torr. Octafluoro-2-butene Pfaltz & Bauer	-45, +29, -710	2 μ A Argon 1 KeV for 1 hour	Better, but only slightly.
SLK 171 B-6-79	1.0×10^{-4} torr. Decafluorobutane but $7. \times 10^{-6}$ torr. resuid. after bake cut	.7 μ A 550.	780 ev.	-704 +400 -900	1×10^{-4} torr. Octafluoro-2-butene Pfaltz & Bauer	-45, +29, -710	2.5 μ A Argon 1 KeV for .5 hour	Same as 169.
SLK 173 B-11-79	Hexafluoroethane Math. Lot #98 1.5×10^{-4} torr. (5×10^{-6})	2.5 μ A 206 Volts	300 ev.	-700 +50 -1200	1×10^{-4} torr. Octafluoro-2-butene Pfaltz & Bauer	-5, +50, -700	10 min. at 2 KeV Argon	5 hr. Target adherent but coming up fast. After stay in vac. sys. one night, gone.
SLK 175 B-11-79 Oil change	Hexafluoroethane Matheson Lot #98 1.5×10^{-5} after Argon purge.	Unable to fire gun with cryobaffles full.						5 hr. Some peel-up visible on removal from vac. sys. Stable 2-3 days. = 2 μ thick.
SLK 177 B-19-79 Complete sys clean up	Hexafluoroethane Matheson Lot #98 1.5×10^{-4} torr. (1.5×10^{-5} after Argon purge)	2.0 μ A 206 Volts	303 ev.	-220 +50 -1400	5×10^{-4} torr. Octafluoro-2-butene Pfaltz & Bauer	-12, +19, -700	45 min. gun gas only. w/o neutrals before start film. 2.5 μ A 206 Volts	
SLK 181 B-21-79	Hexafluoroethane Matheson Lot #98 1.5×10^{-4} torr. (1.5×10^{-5} after Argon purge)	3.05 μ A 224 ev.	271 ev.	-441 +61 -1210	.5 $\times 10^{-4}$ torr.	0, +86, -700 Best focus	30 min. gun gas only. w/o neutrals before start film. 2.5 μ A 206 Volts	5 hr. Star-shaped blotch. Probably amorphous carbon but seems thick.

Table I (continued)

Coat Designation (Notebook Pages)	Gas (Base Pressure) Producer and Lot No. Partial Pressure	Ion Current; Ion Kinetic Energy at Target	Electron Kinetic Energy in Gun Plasma	Extractor Voltage; Anode Voltage; Decel Voltage	Neutral Gas Producer and Lot no. Partial Pressure	Focusing Conditions A, B, C. Sec. Trip.	Target Preparation in Vacuum Chamber	Results
SLK 183 8-28-79	Hexafluoroethane Matheson 1.5×10^{-6} torr.	2.0 μ A 224 ev	271	-441 +61 -1210	$.5 \times 10^{-6}$ torr.	0, +56, -700 Best focus	45 min. gun gas only. W/o neutrals before start film. 2.5 μ A 206 Volts	5 hr. Stable adherent film about 3 μ thick but wrinkled.
SLK 185 9-1-79	Hexafluoroethane Matheson Lot #98 (1.5×10^{-6}) 1.0×10^{-6}	2.0 μ A at 200 ev. 10 hrs. 2.0 μ A at 250 ev. 8 hrs.	250 ev. to 300 ev.	-480 +50, +100 -1000	1.2×10^{-6} torr., 2.2×10^{-6} torr.	0, +50, -700 Defocused	1.5 hr. at 4.5 μ A gun gas.	No film; hole in mirror.

Table I (continued)

phenomenon known to mass spectroscopists (30). In any event, the study was abandoned on communication from Livermore that this approach to film formation was uninteresting.

3

The Fifth Generation Ion Source

Motivated by the need for cleaner vacuum with available equipment, the 6" glass vacuum cross was abandoned in favor of a 4" DependexTM stainless steel vacuum cross. At the same time, a General Electric Monopole 300 was installed to provide residual gas measurements and analysis of ion gun output. The new system provides much better ultimate vacuum since backout was possible without risk of implosion (always a risk with the glass cross). The new vacuum chamber provided ultimate vacuum of 2×10^{-7} torr with cryopumping. Ion source design modifications include introduction of a Helmholtz geometry solenoiding magnet and substitution of a grid cage for the solid anode cylinder used in the fourth generation ion source. These modifications allow easy implementation of differential pumping of the source region with gas flow transverse to direction of ion beam transport.

Materials and Methods

The vacuum chamber employed was a 4" DependexTM type 304 stainless steel cross with a 4" x 2" tee attached as the source chamber. Details of apparatus layout will be discussed in a subsequent section. The chamber was pumped with a 4" CVC oil diffusion pump equipped with a cryobaffle just below the gate valve leading to the main vacuum chamber. Type 704 silicone diffusion pump oil was used as the working fluid.

All ion source structures were assembled from evTM parts produced

by Kimbal Physics, Inc. Metal components were of type 304 stainless steel, while insulating spacers and structural support members were of Al_2O_3 . Emissive filaments were of 0.010" diameter tungsten. Ion source structures were mounted directly on a Dependex 4" diameter vacuum flange via an eV ion optics C 7X7 hole pattern centered on the flange. Gas feed and ion source electrical feedthroughs were provided by a colutron ion source receptacle. The gas feed was electrically isolated by simply not allowing the $\frac{1}{4}$ " feed tube to touch any source parts. The ceramic mount plate used in the fourth generation source was abandoned. The anode cage was fabricated from .06" diameter stainless steel rods cut to 1" lengths and spot welded to a C-7X7-R-1250 eV aperture plate.

Technical grade argon was obtained from Airco while technical grade octafluoro-2-butene was obtained from Pfaltz and Bauer (lot #4548). Research grade normal butane (99.995% pure) was obtained from Phillips Petroleum (lot #1214). The technical grade gases were analysed mass spectrometrically and found to be better than 95% pure.

Apparatus Layout and Experimental Procedure

The layout of the vacuum system and relative positions of the ion source chamber, main chamber, and the monopole mass analyser are shown in Figure 22. The ion source and optical train layout and electrical services are shown in Figure 23. It should be noted that an important change in ion source electrical layout has been intro-

Figure 22. Vacuum apparatus for the Fifth Generation VHY Source

Figure 23. Apparatus electrical layout for the Fifth Generation VHY Ion Source.

Table II

Ion Source	Spot Focused Current Density	Beam Kinetic Energy	Ionic Species
6th generation VHY ion source and soft lander	70 $\mu\text{A}/\text{cm}^2$	50 eV	Octafluoro-2-butene 70 eV electron impact fragments
Colutron ion source	127 $\mu\text{A}/\text{cm}^2$	400 eV	H_2^+
DIDA ion source	3.18 $\mu\text{A}/\text{cm}^2$	200 eV	Ar^+
Hitachi "soft lander"	100 $\mu\text{A}/\text{cm}^2$	70 eV	Ge^+

ced. The voltage divider formerly used to establish anode-cathode bias has been replaced by independent power supplies with floating outputs. This provides a capability of maintaining anode-cathode bias independent of beam kinetic energy, since the bias supplies can be referenced to a single supply used to determine the potential of the entire gun structure relative to ground. The total number of power supplies may appear excessive, but it should be remembered that the layout represents a designer's "bread board" and provides maximum flexibility in experimental work.

Results and Discussion

Ion current focusable through a 1.5 mm aperture at several different total pressures of argon is shown in Figure 24. Comparison of these data with other high yield ion sources (see Table II) shows that at 500 eV final beam energy, the fifth generation ion source produces current densities comparable with commercial high pressure penning and colutron sources but operates with a total pressure of 10^{-6} to 10^{-4} torr in the ionization region, a performance characteristic which allows the generation of high currents of true electron impact fragments of feed gas molecules, as will be subsequently documented.

The importance of the magnetic field at 5×10^{-6} torr argon is shown by Figure 25. After optimization of the ion source and optical train for ion current focused through the 1.5 mm aperture plate the magnetic field is reduced to zero and then increased stepwise to the

Figure 24. Maximum ion current focusable through a 1.5 mm. diam. aperture as a function of pressure.

Figure 25. The effect of axial magnetic field on total ion current.

optimum value and beyond. The electron impact energy here was 70 eV. With no readjustment of focusing lens potentials the data shown as crosses were obtained. However, if the ion current through the 1.5 mm diameter aperture is reoptimized by small variation in focusing lens potentials, the data shown as circles results, and we see a much broader optimum magnetic field region. This result indicates that electron cyclotroning conditions in the ionization region influence ion beam optical properties in several important ways. The most important variable in determining how much ion current can be focused through a small aperture is, however, the voltage placed on the final element of the secondary triplet, i.e. on the beam kinetic energy at the aperture. Figure 26 shows the focused ion current (at two different system pressures of argon) as a function of final stage acceleration for two different magnitude ranges of total ion current. Since the curves are parallel it would appear that space charge is not an important variable at these current levels and since chromatic aberration can be shown to be negligible, spherical aberration is the most likely cause for the observed reduction in focused current density with final kinetic energy. The spot focusing parameter, g_1 , calculated by Harting and Reed for a three-aperture lens similar to the secondary focusing triplet (Figure 23), steadily improves with increasing acceleration. The performance characteristics of the soft lander discussed at length in the next section confirm the spherical aberration hypothesis more completely.

In order to directly compare the ion output of the VHY source with a more conventional low current Nier type source, the faraday cup

Figure 26. Ion current focusable through a 1.5 mm diameter aperture as a function of acceleration voltage in the secondary triplet.

assembly was removed from the fifth generation source structure as shown in Figure 22. In this configuration, ions from the VHY source could be fired through the repeller grid of the Monopole 300 ion source for subsequent mass analysis. (The Nier source filaments were shut down and the repeller grid held at lens C potential for this measurement.) Alternately, simply reconnecting the repeller grid and filaments to the monopole 300 ion source control unit allows collection of mass spectra of the same sample gas with a conventional low yield electron impact ion source. The spectra of research grade normal butane at several electron impact energies is shown in Figure 27, where the fragmentation pattern produced by the Nier source is compared with that produced by the VHY source (the electron impact energy was estimated as the anode, cathode potential difference in the VHY source). Very similar fragmentation patterns are apparent. It appears that the VHY source produces a fragmentation pattern characteristic of a lower impact energy setting for the Nier source. In addition, it should be noted that a marked increase in signal-to-noise ratio is possible with the VHY source (as compared with the Nier source) despite the very poor transfer efficiency of the optics. That is, the VHY source is able to put more ions into the acceptance aperture of a mass analyser than the Nier source, which is standard equipment on the unit, despite a poor optical injection arrangement and great distance between the VHY source and the monopole. With good optics, improvements of 3 orders of magnitude are anticipated from the focusing st. .

The mass spectra of octafluoro-2-butene, the gas used in the soft
lander study to follow, is shown in Figures 28 to 30 where the fragmentation

Figure 27. Mass spectra of N-Butane

Figure 28. Mass spectrum of octafluoro-2-butene
Fifth Generation VHY Ion Source
70 eV electron impact energy

Figure 29. Mass spectrum of octafluoro-2-butene
Monopole 300 ion source at 70 eV

Figure 30. Mass spectrum of octafluoro-2-butene
Fifth Generation VHY ion source
100 eV electron impact energy

produced by the Nier source is again compared with the pattern produced by the VHY gun. It would appear that less fragmentation is produced by the VHY gun at a given anode-cathode setting, and this is in part confirmed by comparison of the spectra shown in Figure 30. With an anode-cathode bias of 100 volts, a mass spectrum is produced which strongly resembles the Nier source spectra above mass 50. The reduced relative ion yield below mass 50 may be due to scattering of low mass ions out of the beam by grazing collisions with neutrals of relatively high mass (remember the VHY source is over 10 cm from the monopole). This phenomenon is under study.

The fifth generation ion source established several important capabilities of the VHY ion source:

- 1) High focused current densities could be produced by ionization at low pressure in a large volume if a relatively high degree of acceleration occurs in first stage focusing.
- 2) True electron impact spectra are produced and, in fact, a reduction in fragmentation is observed.
- 3) The VHY source can improve ion throughput in a typical mass analysis unit even with very adverse injection optics.

The Sixth Generation Ion Source and the Ion "Soft Lander" Experiments

Focusing studies conducted with the fourth and fifth generation ion sources indicated that high focused ion current densities could only be obtained at relatively high final beam kinetic energies with the transfer optics in use. Detailed calculations of lens properties by Harting and Reed (26) suggested that the limiting factor here was spherical aberration in the focusing triplet. Making further use of the Harting and Reed calculations, a decelerating, focusing three element aperture plate lens was designed and built. The triplet lens was designed to reimaged the ion current focused through a 0.25" aperture at 1 to 2 KeV beam energy on a 1.5 mm aperture with a beam energy of 40 to 100 eV with negligible increase in spot size due to spherical aberration. As will be shown below, the design effort was successful.

In addition, several major modifications in ion source design were introduced to make the system more nearly approach ideality.

Materials and Methods

The vacuum station layout was the same as that already described above for the fifth generation ion source. The same Kimbal Physics eVTM parts were used to build all components. Filaments were made from Mackey .010" diameter 1% thoriated tungsten (lot #J299-N) instead of the plain tungsten wire used in the past. Reagent gases were the same as already described for the fifth generation ion source.

Apparatus Layout and Experimental Techniques

The layout of the sixth generation ion source with soft lander is shown in Figure 31. The 2.5" C-CY-1250/2500 cylinder provides a more uniform field in the flight space between the last aperture plate of the secondary focusing triplet and the target aperture. With both the target aperture plate and lens element C held at or near -1 KeV, field penetration from the surrounding vacuum chamber walls had resulted in star shaped ion beam impact patterns near the target aperture in the fourth and fifth generation sources, suggesting that better focused ion currents could be achieved if this particular aberration were removed. Shielding surfaces can, however, be a source of aberration in themselves if insulating deposits are allowed to build up. Experience with the fifth generation source indicated that high focused current density could be obtained at lower pressures, i.e. under cleaner vacuum conditions where insulating deposits develop less rapidly. The only other major change between the fifth and sixth generation ion sources involves the optical arrangement for extraction of ions from the anode cylinder. The conical anticathode "plasma cup" and eV PL C7X7 R 1500 extractor lens were replaced as follows: The anticathode became a simple eV PL C7X7 R 1000 and the extractor was replaced by an eV PL C7X7 R 1250 holding an eV C-CY-1250/1250. The motive here was once again to try to reduce field penetration by shielding the flight path where higher voltages occur. The conical "plasma cup" anticathode was introduced only on the basis of publications involving duo-plasmatron beam quality (30) and offers no real advantage at lower pressures, as will be shown below.

Figure 31. Apparatus electrical layout
Sixth Generation VHY Ion Source and soft lander

Results and Discussion

Using the sixth generation VHY ion source in combination with the ion soft lander structure we have been able to focus 1.2×10^{-6} amps of octafluoro-2-butene fragment ions through a 1.5 mm diameter aperture with a final ion impact kinetic energy of 55 electron volts. This current density was achieved at a total system pressure of 5×10^{-5} torr octafluoro-2-butene without differential pumping of the source region. The ion current cited above was that recorded at an aluminum mirror coating target at the beginning of a film formation attempt. During the subsequent film growth period ion current was observed to decay and could only be corrected by increasing the landing kinetic energy. This would be expected if an insulating layer were being formed from the ions arriving at the mirror surface. In this first low energy coating attempt the measured ion current was allowed to drift to lower values but optimized periodically with adjustment of focusing lens potentials only. In a subsequent study, kinetic energy was increased slowly during the film growth period so that the initial target ion current remained nearly constant for the duration of the growth period. A higher dose should result in a thicker film until beam kinetic energy becomes high enough to cause film mass loss by sputtering. The films formed are adherent and similar in appearance to those formed by the ion induced polymerization technique reported previously. Samples are included with this report.

The ion current density reported above does not necessarily represent a global optimum for this source-optical train configuration. Further,

more detailed optics studies are in progress. Improving electron emission current from the filaments is an especially promising area of development. Thoriated iridium ribbon filaments should prove superior in useful life and emissivity to the thoriated tungsten presently in use. Also, operation at higher ionization region pressures with differential pumping should improve focusable yield. Our next immediate development goal is 5 to 10 microamps through a 1.5 mm diameter aperture at 50 to 100 eV landing energy.

It should be pointed out that the high current density in an area much larger than a microballon is probably more desirable than a similar current density in a 100 micron diameter beam. Beam focusing and steering requirements are less stringent for a large diameter beam which could, in fact, be used to coat several IDF targets at one time. ICF?

The fifth generation ion source was used with the soft lander structure. On comparing the focused current density achieved with the fifth generation ion source-soft lander with the sixth generation ion source-soft lander, an improvement factor of two in focused current density is seen.

A typical "soft lander" coating study begins by cleaning a first surface aluminum mirror with the Lawrence Livermore Laboratory approved method. Then the coater assembly with target is mounted in the vacuum chamber and baked, with cryobaffles filled, for 6 hours prior to beginning a coating run, or until a residual gas pressure less than 5×10^{-7} torr is achieved. At the end of the bakeout period the ion source is turned on and the feed gas pressure adjusted to 5×10^{-5} torr (ion gauge). Lens voltages are then adjusted for optimum ion current.

During the coating runs, ion current is observed to drop slowly as would be expected if an insulating film were building up on the target mirror surface. Ion current levels can be maintained by increasing beam kinetic energy, with some risk of film loss due to sputtering. the two films included with this report were both started with 50 eV landing energy. The film designated SLK II-77 was held at 50 eV for the 5 hour duration of the coating run, during which ion current was observed to drop from an initial value of 1.2×10^{-6} amps to 0.3×10^{-6} amps. In contrast, the film designated SLK II-79 was made with ion current held constant at 0.8×10^{-6} A for the 5 hour duration of the coating run while beam kinetic energy was increased from the starting value of 50 eV to a final value of 90 eV so as to maintain nearly constant ion current.

The ion beam composition for both coating runs is that shown in Figure 30.

Future Directions

The ion source development work discussed in the preceeding pages of this report demonstrates a steady improvement in ion source performance, culminating in a genuine advance in the state of the art. True low to medium energy (40 to 100 eV) electron impact ionization products have been produced at low ion kinetic energy (below 100 eV) and with current densities in excess of $70 \mu\text{A}/\text{cm}^2$. Since ionization occurs at low pressure in a low surface area structure characterized by a low gas residence time, ion-molecule, molecule-molecule, and molecule-surface reaction products do not contribute significantly to ion beam composition as is the case with more typical high pressure sources (10). The mass spectral data presented in the fifth generation ion source section clearly demonstrate that the VHY source is able to produce high yields of reactive, odd electron ions of high mass. Further improvement in source performance is possible, however, and defines the objective of ongoing development work.

Development work is currently directed in four areas.

- 1) Increasing low kinetic energy focused current densities in the ion "soft lander" up to the limits imposed by space charge and chromatic aberration. Improved optics and more emissive filament materials should allow current density approaching $200 \mu\text{A}/\text{cm}^2$.

- 2) Mass analysis of high current ion beams. A 90° sector magnet is presently being configured for beam analysis at 1 to 2 kilovolt beam energies.
- 3) Recent advances in vacuum ultraviolet sources and optics suggest that a high brightness, high current "point source" may be achieved. Desirable optical properties similar to those displayed by the metallic "needle" sources are expected.
- 4) Argon ion beam sputtering of teflon to generate true CF_2 film stoichiometrics at relatively high coating rates. The technique has been reported in the literature (31, 32).

References

1. Wayne Johnson, personal communication.
2. Swinger, "Mass Spectrometer Ion Source with High Yield," J. Appl. Phys. 41, 1946 (1970).
3. Kaufman, NASA Rept. TN-D-585 (1967).
4. Nielsen, Nucl. Inst. & Methods (1957) 1, 289-301.
5. Freeman, "Some General Considerations of Ion Beam Production and Manipulation," in Material Characterization Using Ion Beams, J. P. Thomas and A. Cachard, Eds., Plenum Press, London and New York (1978).
6. Sidenins, "Gas and Vapour Ion Sources for Low Energy Accelerators," in Low Energy Ion Beams, K. G. Stephens, I. H. Wilson and J. L. Moruzzi, Eds., Institute of Physics, Bristol and London, (1978).
7. Septier, "Production of Ion Beams of High Intensity," in Focusing of Charged Particles, Vol. II, A. Septier, Ed., Academic Press, New York and London (1967).
8. Richter and Schwarz, "Chemical Ionization--A Mass Spectrometric Procedure of Increasing Importance," Angew. Chem. Int. Ed. Engl. (1978) 17, 424-439.
9. Morgan, Kelley and Davis, "Technology of Intense Ion Beams," Rev. Sci. Instr. (1967) 38, 4, 467-480.
10. Steinberg, "A Low-Current, Low-Energy Spread Duoplasmatron Ion Source," Rev. Sci. Instr. 42, 3, 354.
11. Anderson, Methods of Exp. Physics (1967) 4, pt. A, 256.
12. Föhrst, "High Frequency Gas Discharge Ion Sources," Angew. Phys. (1958) 10, 546.
13. Nagy, ATOMKI Reports (1963) 5, 143.

14. Beynon and Boyd, "Unimolecular and Collisionally Induced Ion Reactions," in Advances in Mass Spectrometry, N. R. Daley, Ed., Heyden & Sons, London, Philadelphia, Rheine/Westf., vol. 7B (1979), p. 1116.
15. Menzinger and Wahlin, "High Intensity, Low Energy Spread Ion Source for Chemical Accelerators," Rev. Sci. Instr. (1969) 40, 1, 102-105.
16. Dahl, Introduction to Electron and Ion Optics, Academic Press, New York and London (1973).
17. Reader, NASA Rept. NN-D-1163 (1962).
18. Dawson, Quadrupole Mass Spectrometry, Elsevier, New York (1976).
19. Brubaker, "A Study of the Introduction of Ions into the Region of Strong Fields within a Quadrupole Mass Spectrometer," NASA contract no. NASW-1298 (October 1967).
20. Vályi, Atom and Ion Sources, John Wiley and Sons, London, New York, Sydney, Toronto (1977), 128-134.
21. Wilson and Brewer, Ion Beams with Applications to Ion Implantation, John Wiley and Sons, London, New York, Sydney, Toronto (1973), 45-47.
22. Blum and Torrey, "Cold Cathode Quadrupole Mass Spectrometer," Rev. Sci. Instr. (1967) 38, 10, 1404-1408.
23. Yagi, Miyake, Tokuyama, "Low Energy Mass Analysed Ion Beam Deposition," in Low Energy Ion Beams 1977, K. G. Stephens, I. H. Wilson and J. L. Moruzzi, Eds., Institute of Physics, Bristol and London (1978).
24. Amano and Lawson, "Thin Film Deposition Using Low-Energy Ion Beams (2), Pb^+ Ion Beam Deposition and Analysis of Deposits," J. Vac. Sci. Technol. (Mar/Apr 1977) 14, No. 2.

25. Varian area representative, personal communication (price of Varian Extrion Ion Implantation System).
26. Harting and Reed, Electrostatic Lenses, Elsevier Scientific Publishing Co., Amsterdam, Oxford, New York (1976).
27. Reeve, Ph.D: Dissertation, Chemistry Department, University of Illinois (1971).
28. Reinsfelder, Ph.D. Dissertation, Department of Chemistry, University of Arizona (1978).
29. Auger electron spectroscopy provided by Dr. N. A. Armstrong, Department of Chemistry, University of Arizona, Tucson, Arizona.
30. O. B. Morgan, G. G. Kelley and R. C. Davis, "Technology of Intense Ion Beams," Rev. Sci. Instrum. (1967) 38, 4, 467-480.
31. Weismantel, Rost. Proc. 6th International Vac. Congr. (1974), Japm. J. Appl. Physics, Suppl. 2, pt. 1 (1974).
32. Weismantel, Fiedler, Hecht, Reisse, Thin Solid Films (1976) 13, 104.

Appendix I

2-6-80

Ion Soft Lander Experiment for Lawrence Livermore Laboratories

I. Purpose: In order to evaluate Wayne Johnson's estimate of the sticking coefficient for ionic fluorocarbons on aluminum targets ($\alpha = 1.0$) we have to generate $\sim 1 \mu\text{A}$ on a 1 mm diameter area. This is "do-able" at present but only with relatively high ion kinetic energy (see SLK II pp. 31 to 44). The simplest approach is to decelerate and refocus after forming the high current density spot at 500 to 1000 eV. Final impact kinetic energy should be 50 to 100 eV. Under these conditions a growth rate of .1 mm to 1 micron per hour would be achieved if the estimate is correct. Octafluoro-2-butene will be used as the gun gas at ~ 70 eV electron k.e. to provide fairly massive, low velocity projectiles.

II. Decelerator Lens System Design: Based on lens quantities calculated by Harting & Reed, Elsevier Scientific Pub., Amsterdam - Oxford - New York, 1976, Electrostatic Lenses.

Three aperture sys. $A/D = .50$. Figure of merit here is g_2 for $v_3/v_1 = 30$ (Max. in H & R); in accelerating mode g_2 is .4 to .6 for all three element lenses.

$$\frac{v_3'}{v_1'} = \frac{v_1}{v_3} = x, \quad g_2' = x^{-3/2} g_2 \text{ approx. for decelerating lens.}$$

$$g_2' = 10 g_2 \text{ so last } g_2' \text{ is } \sim 4 \text{ to } 6 \text{ for decelerator.}$$

Now, since spot size $\Delta r \approx L (\alpha_0^3) g_2$, where L is object to image distance

in cm and α_0 is the object ray departure half angle (max) from point source on axis, α_0 is in radians. α_0 can be less than 15° from overall geometric considerations and $15^\circ = .26 \text{ rad}$. $(.26)^3 = .0175$ so $\Delta r = .0175(4)(L)$ for 15° and $\Delta r = 6 \times 10^{-4}(4)(L)$ for 5° ; 5° to 7° is most realistic here from considerations of geometry in last section of acceleration trip, which forms first 1 mm spot. If $L < 30 \text{ mm}$ and $5^\circ = \alpha_0$ we have negligible increase in spot size.

$$\text{Cap } f_1 = 1.20$$

$$\text{Cap } f_2 = .79$$

$$\frac{v_3}{v_1} = 30; \quad \frac{v_2}{v_1} = 4.0$$

$$\frac{A}{D} = .5$$

are specs for corresponding Accel. lens. Primed Quant. are for decel. lens required.

$$v_1' = v_3$$

$$x = v_2'/v_1'$$

$$v_2' = v_2$$

$$y = v_3'/v_1'$$

$$v_3' = v_1$$

$$f_1'(x, y) = f_2(x/y, 1/y)$$

$$f_2'(x, y) = f_1(x/y, 1/y)$$

$$P' = Q$$

$$Q' = P$$

P and Q are object and image distances. For decel. lens, $P' = Q$, $Q' = P$.

-C-

$$\frac{Q}{D} = 1.3 \quad m = .4 \quad \frac{P}{D} = 3 \text{ for } m = .4$$

$$\frac{Q}{D} = 3 \text{ for } m = 3 \quad \frac{P}{D} = 2 \quad m = 1.0$$

for Dece1 $P \rightarrow Q', Q \rightarrow P'$

$\frac{A}{D} = .5$ $D = .25"$ for eV plate SS-PL-C5x5-R-250. So for $\frac{A}{D} = .5$
we have a lens spacing of $1/8"$. The layout is shown below.

III. Neutral Particle Impact Rate on Target at Various Pressures:

Object here is to compare neutral impact rate with ion impact rate in
particles/cm² sec units.

$$1 \text{ mA} = 6.52 \times 10^{+15} \text{ ions/sec.}$$

$$1 \text{ } \mu\text{A} = 6.52 \times 10^{+12} \text{ ions/sec.}$$

$$\text{Neutral impact rate} = 2.89 \times 10^{+22} \frac{P}{\sqrt{MT}} \quad \begin{array}{l} P \text{ in torr} \\ M \text{ in daltons} \\ T \text{ in } ^\circ\text{K} \end{array}$$

$$= \frac{P_{eq}}{(2\pi MRT)^{1/2}}$$

at 10^{-5} torr get order of $10^{16} \text{ (cm}^2 \text{ sec.)}^{-1}$

get $\sim 10^{14}$ ions/sec. cm^2 if $1 \mu\text{A}$ through .060" diameter aperture.

So under typical operating conditions likely to have 100 fold excess of neutrals over ions on target. This is noted again for easy reference. First entered in SLK I p.80.

Now calculate coating rate using Wayne's assumption of $\alpha = 1.0$.

IV. Estimate of film growth rate assuming:

- a) density of film $\sim 2 \text{ g./cm}^3$
- b) sticking coefficient = 1.0
- c) 100 Amu is average ion mass

$$\begin{aligned}
 1 \mu\text{A} &= 6.52 \times 10^{12} \frac{\text{ions}}{\text{sec.}} \cdot 100 \frac{\text{Amu}}{\text{ion}} \cdot 1.66 \times 10^{-24} \frac{\text{grams}}{\text{Amu}} = 1.082 \times 10^{-9} \\
 &= 10.82 \times 10^{-10} \frac{\text{grams}}{\text{sec.}} = 5.41 \times 10^{-10} \frac{\text{cm}^3}{\text{sec.}} \\
 \rho &= 2 \text{ g/cm}^3 \Rightarrow .5 \text{ cm}^3/\text{g}
 \end{aligned}$$

Now divide by surface area of impact region.

$$\begin{aligned}
 1) \text{ for .060" diameter hole } &1.8 \times 10^{-2} \text{ cm}^2 \\
 1 \mu\text{A} &= 3.0 \times 10^{-8} \frac{\text{cm}}{\text{sec.}} = 1.80 \times 10^{-6} \frac{\text{cm}}{\text{min.}} \cdot 10^4 \frac{\mu}{\text{cm}} \\
 &= 1.8 \times 10^{-2} \frac{\mu}{\text{min.}} \cdot \frac{60 \text{ min}}{\text{hr.}} = 1.00 \frac{\mu}{\text{hr.}}
 \end{aligned}$$

higher projectile mass $\Rightarrow$ higher coating rate for a given current.

]

Conclusion: There is no need to run longer than 2 hours at $1 \mu\text{A}$. Either it will or it won't. Since these films are insulating, need to worry about grounding actual film surface. Should see fall off in I^+ if film surface is able to stop or repel ions. Now let's do it! With appropriate control

V. Construction Details and Description of Ion Depositer.

Geometry indicated on p. 67 of this notebook reproduced with E.v. parts. Ion source structure same as that shown on p. 53 of this notebook but modified as follows:

- 1) Flight path from lens 3 at sec. trip to aperture plate (.187" diameter aperture) shielded with 1.5" O.D. 2" long eV cylinder. Aperture plate to lens 1 in decelerator shielded by 1" O.D. 1" long e.v. cylinder. For flight space from lens element 3 to target aperture plate have no solid shield but spacers all S.S. tubing. Target mirror isolated from target aperture plate with mica washer cleaned by overnight immersion in MeOH and drying in air 24 hours followed by heat gun ~ 10 min. Electrical contact with mirror front surface established with Tungsten wire lying on mirror side of washer; whole assembly held down with spring clamps. Target cleaned with MeOH and excess removed with lens

paper as per L.L.L. cleaning instructions. Target aperture .060" diameter.

VII. Deposition Experiment

A. System pumped down at 8:00 p.m. 3-12-80 and allowed to pump overnight without cryobaffles. Cryo's filled, 2:00 p.m. Initial pressure 30 min. from start pumpdown 3×10^{-5} . Same next morning. Filling cryobaffles with $L-N_2$ pressure dropped to $\sim 3.5 \times 10^{-7}$ in 15 minutes. Chamber heater at 130 overnight. Now inc. 135 & turn filament on in ion gun at 55 watts for 3 hours. Usual fil-on pressure pulse to $\sim 2 \times 10^{-6}$, then drops. Motor scan spectra at 3:00 p.m. and 10:00 p.m. Major r.g. at $\sim$ Mass 25 on M. meter. See if dec. with time.

Spectra conditions, p. 73

Gun gas - Octafluoro-2-butene, 4548.000640. $P_T = 5 \times 10^{-5}$
for best focusable ion current with deceleration to 70 eV for landing. .403 μA into .060" diameter aperture.

Gun operating conditions

Anode: +41.4 v

Fil.: -55.7 v (15 v 4.25 A 3 μA emission)

Anticathode: -252 v

Extractor: -1800 v

Final: -30.5 v

Sec. Trip.: A: -30.5 v

B: +46.0 v

C: -1000 v

Decelerator Voltages.

L 1:- 1000 volts

have $30 \mu\text{A}/\text{cm}^2$ current
density.

L 2:- 700 volts (740 opt.)

L 3:- 125 volts

Target - 30 volts

Run log.

4:00 current opt. at 200 eV, then dropped target potent to 30 and
reopt. at .409 by 4:30 p.m.

5:00 .412 μA

6:00 .365 μA

6:30 .333 μA adj; by inc anode to 62 v

6:35 .401 μA ; focus in sec. trip.

7:00 .3 x 2 μA

7:30 .412 (82 eV) only set .210 at 40 volt anode. Leave at 40.

8:00 .163 μA Looks like target polarization. Focused; only
gives .177 μA .

Operational note: Under these conditions the magnetic field is only
slightly helpful (only factor at 28% to 15%).

.397 μA with B on - .310 without. Steel cylinder may be perm. magnet

(now-) check it out.

R.G.A. SPECTRA. Operation settings.

Electrometer - 10 m (E.M. on 5)

Recorder - 100 mu f.s., 2 cm/min.

Energy, 3

Focus, 4

Drawout, 7.

Scan 1. 50 ev 3:00 P.M.

Scan 2. 75 ev 3:30 P.M.

Strong water peak indicated --Mass meter needs calibrating.

Total water low enough to ind. go ahead.

8:30 .174 μ A

9:00 .165 μ A at 40 took 80 volts anode to get back to .400 μ A.
inc. anode to 50 eV for next 2 hours. retrim all optics.

9:00 .280 μ A

9:15 .256 & unstable

10:00 .142 & unstable

Target evaluation and conclusions.

Target left in vac. sys. overnight. .2 torr showing by 1:00 p.m.

next day. Brought to atmospheric with Octafluoro-2-butene.

Target coated with ~100 Å AL in SEMQ facility coater. Razor slash
across film as usual. Prior to coat target appears non-uniform--
densest at center with 2 μ thickness if color means anything based
on past experience. Also suspect same or similar composition so

can't completely dismiss ion beam induced polymerization of various neutrals in this system. Need to confirm or deny ion landing mech. Film is adherent. Note also possible outgassing from mica washer. Need to pyro this unit before next run. Suitable control probably Ar^+ beam--this should make no film if true ion lander mechanism operating. The argon expt. will only eliminate system artifacts. Will not eliminate ion induced polymerization of neutrals from gun as possible mechanism of film formation. Need to run several neutral levels with same ion current.

Note: shield cylinder definitely magnetic now--measure cylinder region fields next disassembly, demagnetized with heating. Need magnet characteristic at this point.