A PRELIMINARY INVESTIGATION OF THE THERMAL AND ELECTRICAL BEHAVIOUR OF THIN METAL FILMS

by

DENIS CLYDE BEATTY, B.A., B.Eng.Sc.

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> Department of Engineering Physics McMaster University Hamilton, Ontario, Canada.

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AUTHOR: Denis Clyde Beatty, B.A. (S.G.W.), B.Eng.Sc. (Western)

SUPERVISOR: Dr. J.P. Marton

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#### ABSTRACT

The preliminary investigation of the thermal and electrical behaviour of thin metal films gives evidence, Part I, that several mechanisms are responsible for the change of resistance as the temperature increases from room temperature to 500°C. Firstly, there appears grain growth giving a characteristic decrease in resistance. Secondly, the formation of agglomerates upon the continued growth of grains; especially for the thinner Al and Cr films. This effect tends to increase the resistance and a mathematical model is proposed to explain the results qualitatively. Thirdly, the occur**rence** of what appeared to be an electromigration effect. This latter point provided the incentive for a study on the effects of electromigration in thin aluminum film, Part II. The results of this study are comparable to those obtained by other workers, except that the interpretation for the direction of electromigration in Al is reversed. One possible explanation for the difference in the direction of migration could be due to the interpretation of marker motion. A mathematical model is also proposed for electromigration, in which both the effects due to the applied electric field and the electrons collision with the ions have been taken into consideration. It was found that the effect due to electrons collision with the ions upon the migration of ions could be expressed in terms of an exponential function of the square of the electron to ion collision relaxation time.

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PART I

Annealing Effects on the Resistances of

Al, Cr, Ni, Co, and Fe Thin Films

#### Introduction

Extremely thin metal films in the order of a few angstroms and less do not exhibit normal metallic properties of resistivity. For such thicknesses, the size or mean - free path effect imposes a geometrical boundary restriction on the electron - mobility, and the resistivity for thin films are normally much higher than that of the same bulk material. Such films exhibit negative TCR (temperature coefficient of resistance) and deviate quite widely from Ohm's  $Law^{(1)}$ . These properties are related in part to the occurrence of discreet metallic grains or agglomerates separated by finite gaps that act as barriers to current flow.

Relatively thicker films, such as the ones used in the present work where size effect is not of primary concern, can have their characteristics treated approximately as that of bulk material. A recent paper<sup>(2)</sup> proposes a model of Ni-P film based on Matthiessen's rule which takes into consideration the effects on resistivity of grain growth ( $\rho_1$ ), compositional changes such as the formation of Ni<sub>3</sub>P crystallites ( $\rho_2$ ) and oxidation ( $\rho_3$ ). Therefore, according to Matthiessen's rule:

 $\rho \text{ total} = \rho_1 + \rho_2 + \rho_3.$  (1)

The following set of experiments were an attempt to examine the above proposed model for applicability to five elements, Al, Cr, Co, Ni, and Fe. Only the first term of the model associated with grain growth, however, was being experimentally studied. The other terms associated with impurities and oxidizing mechanisms were controlled for by first, ensuring the five elements were at least 99.995% pure and secondly, performing the experiment in a vacuum annealing furnace at a pressure of about  $10^{-7}$  torr.

### Experiment and Results

The preparation of samples for the experiment consisted of vacuum depositing the five metals onto borosilicate glass rods using standard procedures. These rods were rotated at a constant rate during the deposition in order to ensure a uniform covering. The thicknesses for the five metals were set to give resistance values at decade intervals.

Vacuum annealing of all samples was carried out in the following manner: two freshly deposited resistors were connected to two separate terminals in order that resistance changes could be monitored. The samples were then placed inside a stainless steel tube which was kept evacuated to about  $10^{-7}$  torr pressure. Prior to annealing the tube was flushed several times with helium that had been cooled to liquid nitrogen temperatures, and then reevacuated. The samples were heated from room temperature to 500°C at a constant rate of 5°C/min. with changes to both temperature and resistance being continually monitored.

From the five graphs, plots of resistance versus temperature, as shown in Figures 1 to 5, it appears that for films within the specified thickness and temperature ranges Ni, Co and Fe exhibit a general decreasing resistance. Whereas, in the case of Al and Cr, the increase in resistance is quite pronounced.

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FIGURE 1: Resistance-temperature curves of Al-film during annealing in vacuum.

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Resistance-temperature curves of Cr-film during annealing in vacuum. FIGURE 2:

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FIGURE 3: Resistance-temperature curves of Co-film during annealing in vacuum.

4

-6-



FIGURE 4: Resistance-temperature curves of Fe-film during annealing in vacuum.

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FIGURE 5: Resistance-temperature curves of Ni-film during annealing in vacuum.

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#### Interpretation

Electron diffraction patterns as given in Figures 6 to 8, using the reflectance mode of a TEM (reference voltage 80KV), displayed increased sharpness of rings for the annealed samples. These results seem to indicate that grain growth has taken place upon annealing. In the cases of Al and Cr, the diffraction patterns were less sharp or fewer in number after annealing. Later studies showed that Al and Cr tended to form agglomerates if annealing was prolonged for any length of time at 500°C. Such a structural change would explain the increased resistance; structural changes for thinner films being more pronounced at even lower temperatures, as shown in the graphs.

It appears that, upon annealing these five metal films, the initial film thickness is a predominate factor in dictating the final resistance. The overall reaction will either tend towards grain growth or the formation of agglomerates. For Al and Cr the graphs show that for thicker films the initial trend is towards a decreased resistance. As the temperature continues to increase, the grain growth mechanism gives way to film thinning and eventually to the formation of agglomerates. In order to account for the effect on resistance due to this later process, a mathematical model is proposed (for a detailed derivation, see Appendix-Part I).

The model depicts the metal film as growing semispherical globs or islands. As the temperature increases further from room temperature, these metal globs become more pronounced with less and less material making the interconnections, as shown in Figure D of Appendix-Part I. Based on this model, the total resistance of the film can be expressed as

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## FIGURE 7: Diffraction patterns of Nickel.





FIGURE 8(b):

Diffraction patterns of Chromium annealed at 500°C.

$$R = \frac{C2\rho}{\pi a} \ln(\frac{2a - \Delta X}{\Delta X})$$
(2)

where R is the final overall resistance of the film,  $\rho$  is the resistivity of the metal, a is the radial distance of the agglomerate,  $\Delta X$  is a temperature dependent variable, and C is a proportionality constant for a given film.

A plot of equation (2) is given in Figure 9. Comparisons between Figure 9 and 2 show a noticeable similarity between these curves indicating that the physical model is indeed reasonable.

This concept of semispherical formation is probably a reasonable beginning towards a model. Since the spherical configuration is of minimum free energy, it seems quite natural to assume that the formation of semispherical globs in the film as the temperature increases.

Further studies lead to a finding of what appeared to be an electromigration effect. At the instant when agglomeration appears to predominate, it was observed that the increasing resistance could either be enhanced by continually monitoring the resistance change or inhibited by intermittent monitoring. The resistance recording device used in all experiments was a Radiometer constant current, battery operated electrometer Type GVM 30a. Being a constant current device, the current density through the sample could increase at the grain boundaries. This in turn could accelerate the thinning processes of agglomeration through the effects of electromigration, which could explain the accelerated increased resistance. The appearance of electromigration initiated the research for Part II of this report.



FIGURE 9: A plot of Equation (7) by using arbitrary numerical constants.

### Conclusion

The results and corresponding interpretation of this portion on thermal and electrical properties of the five metals are only preliminary. From the results and associated graphs, it appears that the changing values of resistance can be associated with three predominate mechanisms that go on within the film as the temperature increases from room temperature to 500°C.

The first of these mechanisms is grain growth, providing a decrease in resistance. This decrease is quite pronounced for the films of Ni, Co, and Fe, and much less for the films of Al and Cr within the specified range of thicknesses. Secondly, there appears to be the formation of agglomerates which a proposed mathematical model attempts to illustrate as the predominate factor attributing to the increased film resistance of the Al and Cr samples. It is expected that similar results would have occurred for the metals of Ni, Co, and Fe if much thinner films had been tested. A third mechanism which can contribute to increased resistance is that of electromigration. It was suspected that this was the mechanism occurring within the latter stages of increased temperature for the Al and Cr films. Further studies are needed in order to get a better understanding of these mechanisms which contribute to increased film resistance as a function of increased temperature.

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## PART II

Electromigration of Aluminum

in Thin Aluminum Film

#### Introduction

Electromigration is the phenomenon of mass transport of material resulting from the passage of a dc current. It has been identified as the cause of failures in thin film aluminum conductors, as used in planar silicon devices.

The effect of electromigration is quite similar to diffusion mechanisms. One such mechanism is the Soret effect, which can be observed simultaneously with electromigration. In this case, mass transport occurs between two regions of a sample which are maintained at different temperatures. Soret effect is therefore known as thermal migration. It can readily be distinguished from electromigration by conducting simultaneous studies with ac and dc currents, yet maintaining the same current densities.

For thin films, electromigration phenomenon is apparently not significantly different from the phenomenon which have been observed to occur in the bulk samples. Over the range of temperatures which are of interest for electromigration in films, mass transport does not occur through lattice diffusion but proceeds along the grain boundaries. This is more likely to happen in thin films where grain size is small and the boundaries are abundant. Of course lattice and grain boundary diffusion always occur simultaneously in polycrystalline materials. However, as a rule it is the most rapid mechanism which contributes most to the transport phenomenon.

The above theory on electromigration in aluminum thin films has been the object of numerous publications (3-30). The values of activation energy which have been determined, at temperatures up to 350°C, are of the order of 0.5 to 0.6 eV(10,13,16,22,25). These magnitudes of the activation energy support the theory that transport would occur through grain boundary diffusion since lattice diffusion would give a much higher value about 1.4 eV. This latter point was confirmed by an experiment<sup>(21)</sup> which shows that single crystal aluminum thin film conductors are practically uneffected by conditions of current density and temperature which usually cause rapid failure in polycrystalline films.

For all preceding work involving aluminum film, the direction of migration has always been interpreted as moving towards the anode (18,31,32). The present researchers have observed just the opposite effect, that migration appears to proceed towards the cathode.

An extensive review on electromigration in bulk metallic samples was published in 1963<sup>(33)</sup>. Other works since 1963 are given in References 34 to 49. In these works the total force in the metal upon the moving atoms is conceived as the resultant of two forces which are often of opposite signs. The first force,  $F_c$ , is thought of as resulting from the exchange of momentum between scattered charge carriers and atoms<sup>(34)</sup>. It is therefore the force which has the same direction as the motion of charge carriers and, for this reason, is sometimes referred to as the electron wind force. Theoretical treatment indicates the  $F_c$  is inversely proportional to the resistivity of the metal and, therefore, it decreases with increased temperature. The second force,  $F_E$ , is due to the interaction between the ionic core of the atoms and the applied field; it is assumed proportional to the product of the field strength (E) and the valence (Z) of the metal. Now all previous derivations give this force upon the ion, as being directed towards the negative electrode, and primarily independent of temperature. However, in

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the work by Wever<sup>(33)</sup>, page 338 notes that the migration in copper reverses direction from anode to cathode when the temperature is between 900°C and 1000°C. With respect to these results by Wever and our present findings, it is proposed that  $F_{E'}$  is also temperature dependent, in which  $F_{E'}$  is the effective force experienced by the ions. The probability of an ion jump in the direction  $F_{F'}$  is given as

$$F_{E} = \beta' \exp(-\Sigma m - \frac{ZeE'a}{2} + \frac{Z\alpha\omega\Sigma}{2})/kT$$
(3)

where  $\Sigma m$  is the height of the potential barrier,  $\beta'$  is a constant and is related to the ratio of vacancies to ions and the vibrational frequency, Z is the valence, e is the electron charge, E' is the effective field as seen by the metal ions, "a" is the lattice spacing,  $\alpha\omega\Sigma$  is the energy acquired by the ion resulting from collision with electrons, k is Boltzman's constant and T is the absolute temperature. In the F<sub>C</sub> direction, the probability of an ion jump is given as

$$F_{C} = \beta' \exp(-m + \frac{ZeE'a}{2} - \frac{Z\alpha\omega\Sigma}{2})/kT$$
(4)

When the last term is much smaller than the second last term in the bracket, which is apparent in our case, then the drift velocity of ion migration can be written as

$$\overline{V} = (F_E - F_C)a = \beta' \frac{ZeE'a^2}{kT} \exp(-\frac{Q}{kT})$$
(5)

where Q is the activation energy. Refer to the Appendix-Part II for a more complete derivation.

Another query of former studies, which the present researchers have difficulty in interpreting, is marker mobility. Based upon Huntington's works et al<sup>(34)</sup> and a subsequent article<sup>(41)</sup>, the marker on the metal surface is assumed to be similar in nature as voids within the metal. Therfore, explanation as to the direction of metal mobility is based solely upon that assumption; if the marker is a void and it moves to the cathode then presumably metal has been displaced towards the anode. These same conditions for marker studies were attempted with the present experimental set-up. However, any similarity of interpretation to the other researchers' findings was not very explicit. Either the markers, upon annealing, were absorbed and vanished, exhibiting no trend of mobility towards either electrode, or else they appeared to be displaced with the metal. Further elaboration on marker studies is outlined in the conclusion.

### Experiment and Results

The aluminum used in these experiments was given as 99.995% pure. A 5.0 mm wide X 15.0 mm long strip of the metal was vacuum evaporated onto a quartz substrate using standard preparative techniques. The metal strip was then clamped between two electrodes. An identical procedure to that described in Part I was used to mount the quartz substrate in the vacuum furnace. Temperature to the furnace was preset to a desired temperature and automatically maintained at that temperature to within  $\pm 5^{\circ}$ C limits.

D.C. current to the sample was supplied by a 12 volt car battery. The current was preset before the circuit was completed. Values of current, initial and final resistance at the preset temperature, and the time to rupture were the parameters recorded.

Tables I and II give the observed data of the calculated values of velocity  $\overline{V}$  and the parameter  $\beta'$  for the two different currents and thicknesses. The value of S, distance, was interpreted as being one-half the distance over which the migration effect can be observed (see Figure 12). Note that for all samples and over all temperature ranges used in these experiments, migrations takes place from the anode towards the cathode (see Figures 10 to 12). The effect is more readily observed by using the transmission mode of the optic microscope (see Figure 14). Here the surface of migrating metal is opaque when compared to the surrounding matrix of metal and voids.

Repeating the experiment, but this time testing for the Soret effect by reversing the potential to the electrodes, gave identical results. Here, at least was an indication that thermal migration was not the predominant factor giving rise to the characteristic patterns.

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### TABLE I

Electromigration of 1000Å Aluminum Film at Constant Temperatures With an Initial Current of 3.0 Amperes

	. Temp °K	S(mm)	t(sec)	V(m/sec)	R <sub>f</sub> -Ω-T°K	β'(10 <sup>15</sup> sec)	
1	773	2.45	3.0	9.8 × 10 <sup>-4</sup>	3.0	2.076	
2	723	2.25	4.5	5.0 x $10^{-4}$	3.1	1.854	
3	673	2.10	11.5	1.9 x 10 <sup>-4</sup>	2.0	1.795	
4	623	2.00	14.2	$1.4 \times 10^{-4}$	2.4	2.808	
5	573	2.05	28.0	$0.73 \times 10^{-4}$	2.4	3.586	
6	523	1.80	52.4	$0.35 \times 10^{-4}$	2.4	4.935	
7	473	1.65	61.8	$0.27 \times 10^{-4}$	2.2	14.047	
8	423	1.75	80.6	$0.21 \times 10^{-4}$	2.4	58.064	
9	373	1.80	355.0	$0.05 \times 10^{-4}$	2.2	107.466	

### TABLE II

Electromigration of 2500Å Aluminum Film at Constant Temperatures With an Initial Current of 5.5 Amperes

	Temp°K	S(mm)	t(sec)	V(m/sec)	R <sub>f</sub> -Ω-T°K	β'(10 <sup>15</sup> sec)
1	773	3.90	2.8	13.9 x 10 <sup>-4</sup>	1.4	4.484
2	673	2.25	3.7	$6.0 \times 10^{-4}$	1.2	6.574
3	573	1.80	8.2	$2.2 \times 10^{-4}$	1.1	12.544
4	473	2.05	22.8	$0.9 \times 10^{-4}$	1.0	54.544
5	373	1.85	53.0	$0.3 \times 10^{-4}$	0.8	958.40



Anode

FIGURE 10: Electromigration of Al-film (reflection). The result was obtained by increasing the sample temperature linearly from room temperature to 450°C.



FIGURE 11: Top of Figure 10 magnified 225 times.



Anode

FIGURE 12: Electromigration of Al-film (reflection). The result was obtained by keeping the sample at a constant temperature of 450°C.



FIGURE 13: Top part of Figure 12 (reflection) magnified 500 times.


Comparative studies were again tried using alternating currents (Figures 15 and 16). The results presented show a very symmetrical design about the point of rupture without any preferential buildup of material towards either extreme. Another interesting find from this experiment was that the RMS current density needed for rupture seems to be higher than that required for dc currents.



FIGURE 15: Effects of alternating current on Al-film, 75X using transmitted light.



FIGURE 16: Effects of alternating current on Al-film, 75X using reflected light.

#### Interpretation

The graphs in Figure 17 are similar to an Arrhenius plot of diffusion coefficients for both lattice (single crystal) and for grain boundary diffusion<sup>(50)</sup>. Such a plot consists of the diffusion coefficients plotted logarithmically against the reciprocal of the absolute temperature. The concept being that as the grains become larger, the polycrystalline curves should approach asymtotically the curve of the single crystal at a much lower temperature.

The difference in the intercepts which these two curves make with the velocity axis is due in part to the difference in current densities. For higher current densities, it could be expected that the velocity would increase more rapidly as the higher temperatures are approached. Some evidence of this effective mechanism is provided in Figure 18.  $\beta'$  at the lower temperatures increases rapidly, having a very steep curve, whereas at higher temperatures it approaches more nearly a single value. The actual value of  $\beta$ , referring to the appendix, is the ratio of vacancy to lattice frequencies. It may therefore be at lower temperatures where there is an abundance of vacancies in the freshly deposited film and which, in surrounding the metal ions, influences the vibrational frequncy of the ions. However, as the temperature is increased, the number of vacancies is reduced through the effects of annealing. The metal ions are less affected by the remaining vacancies and their vibrational frequency approaches more closely that of the perfect lattice. Hence,  $\beta'$  becomes closer to the single value of the vibrational frequency for an ion situated in a perfect lattice.





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<u>FIGURE 18</u>: A plot of the parameter  $\beta'$  appearing in Equation (5) against sample-temperature T.

As to the phenomenon of reversing migration when temperature changes, such as in the case of  $Cu^{(33)}$ , it is proposed that the effect is primarily due to two reasons:

(1) Since the electron-wind effect is inversely proportional to the square of resistivity (see equation (14) of Appendix-Part II) as temperature increases this effect decreases.

(2) As temperature increases, the screening effect on the ions decreases (54), hence, increasing the effective field strength as seen by the metal ions.

Then, when the last two terms in the brackets of equation (14) become comparable, then migration could switch directions. Consequently, migration to either (<sup>cathode</sup> to anode anode) is possible and depends upon the magnitude of these last two terms in the proposed mathematical model.

#### Conclusion

Our results, except for the direction of migration, are quite compatable and with the same order of magnitude as those of other workers. It is the direction of migration which remains the point in question. It is apparent from the results displayed in Figure 14 that the metal has indeed become thicker in the direction of the cathode. That the area near the anode becomes transparent with an abrupt crest shape configuration separating substrate and metal film.

From graphical recordings of the applied current we know that the electric current is not constant throughout the experiment. There appears to be an initial slow exponential decay before a sudden breakdown. However, the applied electric field E was assumed to be constant even though the current decreased. This assumption was justified on the basis that as J, the current, decreases also the film becomes thinner due to the migration of metal and therefore,  $\rho$ , the resistivity increased.

Perhaps the greatest single difference between this piece of research, compared to others associated with aluminum thin film is in the presentation of samples within a temperature controlled environment. For this research the samples are placed in a controlled environment with a uniform  $\pm 5^{\circ}$ C temperature gradient extending beyond the sample area. In a review article<sup>(51)</sup>, the technique used extensively by Huntington and co-workers was to use the same electrical current in the study of migration to heat the specimen. This results in a temperature gradient where the temperature is at a maximum in the middle of the specimen and much lower at one end, which is apparently water cooled. Though this technique has the

advantage of providing the marker velocity at many temperatures from just one sample, it will also produce marker velocity through the Soret effect. The Soret effect will produce a positive marker velocity on one side of the plane of maximum temperature and a negative marker velocity on the other side.

Marker techniques, as reported, suffer from a number of experimental difficulties in addition to the Soret effect. There are other effects which come into play as specimen creep due to surface tensions, sagging or shrinking of the specimen, specimen length change and local overheating due to void formation, surface diffusion, evaporation and condensation, and changes in the specimen's dimension in the transverse direction can produce apparent marker shift.

A much better technique to overcome most difficulties with the marker studies has been used by Gilder and Lazarus<sup>(52)</sup>. The technique uses two radioactive isotopes, one of the element whose motion is to be studied, and the other of a material which behaves as an inert marker in the matrix. After a current has been passed through the sample for a given time, then the relative distance "S" between consentrations of the trace element to that of the marker gives a direct measurement of the velocity "V", independent of dimensional changes. Further experiments on electromigration, as presented here, could use the radioactive tracer technique towards a better understanding of the mechanics involved in the mobility and the parameters used to define the proposed mathematical model.

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#### APPENDIX - PART I

The model used to formulate the mathematical expression relating increased film resistance to the formation of agglomerates is diagramatically outlined in the set of Figures A to D. When the metal film is freshly deposited, it is pictured as a continuous layer, Figure A. As the temperature increases from room temperature on up, in this case 500°C, the film begins to shrink and nucleate into grains at preferred sights. These sights develop, growing apart from neighbouring sights to form islands, Figures B to D.

Figure E shows one island and the mathematical parameters used to describe the dimensional growth of the agglomerates. Looking down onto the film, Figure F, it can be pictured as rows of spherical islands whose centres are situated an equipotential distance apart. The increasing film resistance is seen to occur at those points of intersect between spheres in the direction of current flow. An equivalent series-parallel resistance network relates this discrete intersect resistance, r, to the total circuit or thin film resistance R.

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# SIDE VIEWS

OF THE THIN METAL FILM

(D) EVEVER EVEN

AGGOLMERATES



SIDE VIEW OF AN AGGLOMERATE

=  $C \frac{2\rho}{\pi q} \ln \left[ \frac{2q - \Delta X}{\Delta X} \right]$ (4)

#### APPENDIX - PART II

For an electron of charge e in an electric field E, the drift velocity v can be written as

$$v = \frac{eE\tau}{m}$$
(1)

and the corresponding energy can be given as

ε

then,

$$=\frac{1}{2}mv^2$$
 (2)

$$\varepsilon = \frac{e^2 E^2 \tau^2}{2m}$$

where m is the mass of the electron and  $\tau$  is the relaxation time of collisions between electron and the positive ions.

During one collision, the electron will transfer a fraction of its energy  $\alpha \varepsilon$  to the ion, where  $\alpha \leq 1$ . If there are  $Z_{\omega}$  collisions between the electron and the ion during the time interval in which the ion makes one jump over the lattice potential barrier, then the total energy transferred to a given ion resulting from the collisions can be expressed as

Ζαωε (3)

Now the jumping probability of an ion, which has acquired the energy  $Z_{\alpha\omega\varepsilon}$  in a current carrying metal moving against the applied field E, is given as;

$$f_{c} = \frac{n}{n'} v' \exp(-\varepsilon m + \frac{ZeE'a}{2} - \frac{Z\alpha\omega\varepsilon}{2})/kT$$
(4)

In the direction of the field the jumping probability is given as;

$$f_{E} = \frac{n}{n'} v' exp(-\varepsilon m - \frac{ZeE'a}{2} + \frac{Z\alpha\omega\varepsilon}{2})/kT$$
(5)

where n and n' are the densities of vacancies and positive ions where n + n' = constant for the system in question, v' is the vibrational frequency of ions under consideration,  $\epsilon m$  is the lattice potential barrier height, Z is the valence of the positive ion, E' is the effective field as seen by the ion as a result of electron screening, a is the lattice constant, k is Boltzman's constant, and T is the absolute temperature. Then the net jumping probability is

$$f = f_c - f_E$$
(6)

$$f = \frac{n}{n'} v' \exp(\frac{-\varepsilon m}{kT}) \{ \exp(\frac{ZeE'a}{2} - \frac{Z\alpha\omega\varepsilon}{2})/kT - \exp(\frac{Z\alpha\omega\varepsilon}{2} - \frac{ZeE'a}{2})/kT \}$$

For the case where

$$Z_{\alpha\omega\varepsilon} \ll ZeE'a$$
 (7)

$$f = \frac{n}{n'} v' \frac{ZeE'a}{kT} exp(\frac{-\varepsilon m}{kT})$$
(8)

From Reference (53)

$$\frac{n}{n'} = \left(\frac{v}{v'}\right)^{3x} \exp\left(\frac{-\varepsilon v}{kT}\right)$$
(9)

where  $\varepsilon v$  is the activation energy required to form a vacancy, x is the number of nearest neighbours, and v is the vibration frequency for an ion situated in a perfect lattice. Combining equations (8) and (9)

$$\mathbf{f} = \left(\frac{\nu}{\nu^{T}}\right)^{3x-1} \nu \frac{ZeE'a}{kT} \exp\left(\frac{-2\varepsilon m + \varepsilon\nu}{2kT}\right) = \beta \nu \frac{ZeE'a}{kT} \exp\left(\frac{-Q}{kT}\right)$$
(10)

where  $\beta = \left(\frac{\nu}{\nu^{-1}}\right)^{3x-1} > 1$  for an imperfect crystal; x = 12 for the fcc structure, reference (53); and Q =  $\varepsilon m + \frac{\varepsilon \nu}{2}$  is the activation energy. The drift velocity of an ion's migration is

$$\bar{\mathbf{v}} = \mathbf{fa} = \beta' \frac{ZeE'a^2}{kT} \exp(\frac{-Q}{kT})$$
(11)

where  $\beta' = \beta v$ .

For the case where ZeE'a is in the same order of magnitude as  $Z_{\alpha\omega\epsilon}$  , then

$$\bar{\mathbf{v}} = \frac{\beta'}{kT} \exp(\frac{-Q}{kT}) \{ ZeE'a^2 - Za\alpha\omega\varepsilon \}$$
(12)

Now

$$\varepsilon = \frac{e^2 E^2 \tau^2}{2m} = \frac{E^2 m}{2N^2 \rho^2 e^2}$$
(13)

where N is the electron density and  $\rho$  is the resistivity of the metal. Substituting equation (13) into equation (12), the final form for the velocity of migration is given as

$$\bar{\mathbf{v}} = \frac{\beta'}{kT} \exp(\frac{-Q}{kT}) \{ ZeE'a^2 - \frac{Za\alpha\omega E^2m}{2N^2\rho^2 e^2} \}$$
(14)

where the ZeE'a term depicts migration towards the cathode due to the applied electric field and the  $\frac{Za\alpha\omega E^2m}{2N^2\rho^2e^2}$  depicts migration towards the anode due to the electron wind effect.

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DESIGN AND CONSTRUCTION OF THE 150 kV-ION IMPLANTATION ACCELERATOR SYSTEM WITH A CRYOGENIC EXPERIMENTAL CHAMBER"

by

DENIS CLYDE BEATTY, B.A., B.Eng.Sc. PART B: MCMASTER (ON-CAMPUS) PROJECT\*

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AUTHOR: Denis Clyde Beatty, B.A. (S.G.W.), B.Eng.Sc. (Western)

SUPERVISOR: Dr. D.A. Thompson

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#### INTRODUCTION

Currently the most important and widely used application utilizing ion beams, especially from an industrial point of view, is the doping of semiconductors. This process involves the injection of selected ions to a desired depth and concentration into the semiconductor crystal. Such as, the forming of well defined complementary substrates with controlled resistivity (C-MOS devices). The fact that dopant characteristics of a substrate can be well defined compared to diffusion techniques reduces the possibility of gate overlap. The reduction of gate overlap reduces the capacitance between the drain and the source, which in turn improves the switching speed and reduces cross-talk and switching noise. Another application which utilizes the doping accuracy are linear resistors. These resistors are used in resistor-ratios for digital to analogue converters; also as load devices for linear and digital circuits. Other applications, just in the mentioning, are the uses of ion implantation for radiation hardening, formation of high voltage and microwave devices, and for application to controlled field inversion. Using low-energy ion implantations, controlled, ordered and disordered reactions of a lattice can be undertaken. With the introduction of interstitial foreign atoms into the crystalline matrix the chemical reaction rates at surfaces can be significantly altered. Ions when deposited on or just beneath a surface can passivate the surface, reducing its susceptibility to chemical reactions. As an example,  $SiO_2$  and  $Si_3N_4$  passivation layers have been

grown by the bombardment of silicon surfaces with intense beams of lowenergy oxygen and nitrogen ions.

In the following report, a description is given of an ion-source and accelerating system mounted in line with a 3 MeV Van der Graaf (KN) accelerator. Using MeV,  $He^+$  and  $H^+$  particles, channeling-effect measurements can then be performed in situ. on the freshly implanted substrate. Such a set-up, where the sample remains in one location throughout the entirity of a channeling experiment should permit a more accurate quantitative measure on the amount of disorder created by the implanted ions.

These are but a few applications of an ion beam. The ion source being the most important single element in an ion-beam system. The characteristics of the source determines to a great extent the performance of the remaining systems. For this reason a considerable amount of developmental effort has been expended over the past thirty or more years to evolve improved types and embodiments of ion sources.

The first part of this report will describe the basic mechanisms underlying the "Danfysik" hot cathode type ion source giving advantages and disadvantages of this source. The second part describes those parameters necessary in getting a uniform (stigmatic) beam down to its target area. Lastly, the third section describes a cryogenic experimental chamber that permits the implantation of samples at temperatures varying from 40 to 700 degrees Kelvin.

#### PART I

### ELECTRON IMPACT IONIZATION PHENOMENA

Positive ions can be produced in four possible ionization processes, namely,

Electron impact ionization,

Surface ionization process,

Ion impact ionization (sputtering),

Charge exchange.

The Danfysik ion source, which is the primary concern of this report, uses the first of these ionization processes.

The electron impact ionization process is probably the most usual and commonly applied technique in ion sources. This is simply the transfer of sufficient potential energy to an atom on impact by a moving electron so that the atom is ionized. The technique is used in several ways, defining the type of ion source as, in an arc discharge, in a confined electron cloud (Danfysik), in an rf discharge, and in a spark. The electron cloud ion source configuration is the arc, which will be described in most detail later in this report.

The efficiency of ionization<sup>(1)</sup>, or the actual number of ionizing collisions suffered by an electron in passing through a gas per unit path length per unit pressures is called the differential ionization coefficient  $S_e$ . This coefficient depends upon the electron energy  $K_o = m_e V_e^2/2$  for various elements (Figure 1); where  $m_e$  is the mass of the





electron and  $V_e$  is the velocity of the electron after impact with an atom. Now for ionization to occur  $K_o \ge eV_i$  where  $V_i$  is the internal potential energy of the atom. For example, the energy required to detach the electron from a hydrogen atom, which has one positive nuclear charge (Z = 1) is given by the sum of the potential and kinetic energy,

$$eV_{i} = \frac{Ze^{2}}{r} + \frac{mv^{2}}{2}$$
 (1)

Consequently, the initial kinetic energy of an incident electron can be converted into potential energy of an atom in a single inelastic collision, and an electron can be released from an atom when the kinetic energy of the hitting electron exceeds the ionization energy of the atom. Hence an electron with a smaller energy cannot ionize the atom and so the probability of ionization is zero for  $V < V_i$ , V being the potential energy of the electron after conclusion. On the basis of Bohr's theory, the ionization energy is a sharply defined quantity, and therefore it would be expected that the ionization probability curve would rise sharply from zero at  $V = V_i$ . Figure 1 of ionization efficiency is essentially a measure of the Bohr probability. Usually this is defined for positive ions; where one incident electron produces per cm of path time at 1 mm Hg and 0°C, one electron and a singularly charged positive ion . The value of ionization efficiency is also numerically equal to the ionization cross section.

From the curves of Figure 1 it can be seen that the electrons with energies much less than or much greater than the atomic and molecular ionization energies do not ionize those particles very effectively.

Se lies for the majority of gases, so far investigated between about 80 and 120 eV. The exception being the alkali vapours whose maximum is between about 15 and 30 eV. Consequently, electron energies a few times the ionization energies are optimum for electron impact ionizations.

When the energized electron loses energy from ionizing and scattering collisions, its ionizing effectiveness varies. Energetic electrons can easily cause efficient ionization if they can be maintained in the ionizing volume for a time long enough for them to lose essentially all their energy by collisions. External restrictions can be induced by confining the electrons to gas interaction by either volumetric restrictions or from a magnetic field, or both. These techniques contribute to larger path lengths and electron lifetimes for optimizing the ionization efficiency. Consequently, referring to Figure 1, if the initial energy of the electron is  $K_0 \leq z$  eV then von Engel's "Differential ionization coefficient Se" can be approximated as

$$Se \simeq a(eV - eV_i) . \tag{2}$$

Therefore, as the primary or "knock-on" electron loses energies, the ionization efficiency or cross section of the atom increases to the optimum value "Se" and then its energies fall off until the electron no longer causes ionization. For higher states of ionization, Bleakney<sup>(2)</sup> showed the the electron energy required increased quite significantly. That is, using mercury, the multiple ionization energies were reported to be:

<u>BLE I</u>	
Energy Range (eV) K <sub>o</sub>	
10-16	
29-50	
71-150	
143-200	

Electron impact ionization is conceptually a very simple technique for creating ions as well as being the most effective and flexible of methods. The description of how Von Engel's theories are made applicable to the design of ion sources is given in the work by Cobine<sup>(3)</sup>.

The Danfysik hot cathode or thermionic emitter type source, being the one currently employed, can be subdivided down into its various integral parts as:

1) Thermionic emitter,

2) Ion gun assembly,

3) Ion extraction assembly.

Using these as subtitles, a design analysis can be reconstructed on paper of the present system bringing to light methods for optimizing the source efficiency and improvements upon the present engineering.

## Hot (Thermionic) Emitting Systems

This is the system which can use externally heated cathode in contrast to ion bombardment or cold cathode assemblies. Figure 2 illustrates diagramatically the potential and electric field versus distance in an arc with electrons furnished by a hot emitter.



Figure 2: Potential and electric field versus distance in an arc with electrons furnished by a hot (thermionic) emitter<sup>(4)</sup>. The electric field E in the sheath region shows the typical space-charge-limited emission characteristics, that is, curving into zero field at both the cathode and sheath sides. This occurs because both surfaces are space-charge-limited emitters. The potential (V) rises rapidly near the cathode surface and remains constant (V<sub>d</sub>) the length of the plasma. There is a slight increase in both the field and potential near the anode which reflects the existence of a thin sheath (anode) at this boundary surface. The normal value for a potential drop across the sheath is in the order of 10 to 20 V. Plasma ion density values are typically in the range of  $10^{12} - 10^{14}$  ions/cm<sup>3</sup> for neutral atom densities, which corresponds to a pressure in the range of  $10^{-5}$  to  $10^{-3}$  torr.

Now the voltage applied between cathode and anode creates a region of space-charge-limited potential forming the cathode sheath. As illustrated in Figure 2, electrons are accelerated through this space-charge-region and in turn, ionize the gas in the interaction (plasma) region, where they form ions and secondary electrons. If the gas pressure is not too high, the electrons will flow from the cathode to the plasma as if it were the anode in a space-charge-limited diode. The corresponding electron current density<sup>(3)</sup> is given by the Child-Langmuir equation

$$J_{-} = \sqrt{\frac{z_{e}}{m_{e}}} \frac{V_{d}^{3/2}}{9\pi d_{s}^{2}} , \qquad (3)$$

where  $V_d$  and  $d_s$  represent the potential drop and thickness of the sheath

which is assumed to be geometrically planar. The ions will flow in the reverse direction as if the plasma were an ion source under the influence of a space-charge-limited emission condition. This ion current density is given by:

$$D_{+} = \sqrt{\frac{z_{e}}{m_{i}}} \frac{V_{d}^{3/2}}{9\pi d_{s}^{2}} , \qquad (4)$$

where m; is the atomic mass associated with the ion.

Now the ratio between these current densities is given by

$$\frac{J_{-}}{J_{+}} = \sqrt{\frac{m_{i}}{m_{e}}} , \qquad (5)$$

such that the electron current density is about roughly 200 times that of the ions. The sheath in its itself is not a neutral region since the electric field accelerating the electrons and ions, such that the potential of that region near the plasma, is determined by an ion space charge whereas the rest of the sheath is influenced predominantly by an electron space. The glow which can be observed around the discharge region is due to the small amount of recombination of the slower ions and electrons corresponding to the low energy portion of the von Engel curve (Figure 1) called a glow discharge.

If the neutral gas supply is less than a limiting value to maintain the critical pressure associated with the plasma, the rate of ion density formation in the plasma column will not be sufficient to supply the necessary ion current to maintain the discharge. Should this happen,
the arc becomes a glow discharge or is extinguished.

Positive ion density can be further increased by applying a uniform magnetic field which causes a charge particle, moving perpendicular to the magnetic, to follow a circular path. Cobine<sup>(3)</sup> gives a detached description of the charge particle motion about a circular path with the radius of the circle given as

$$r = mV/qB , (6)$$

where V and B are the velocities of the particle in the applied magnetic field strength. This resulting helical path causes the electrons to remain in the discharge region for long periods of time, which increases the probability of ion formation per charged particle, and thus increasing the ionization efficiency of the source.

### Ion Gun Assembly

Most Hot Cathode Electron impact mass assemblies consist of a cylindrical ionization chamber with the gas introduced from the end or sides, an electron source and extraction electrode.

Figure 3 shows the Danfysik system with the gas or vapour internal input port located towards the anode end, cathode and filament centrally located, and a first extraction port located at the other end. The central hole arrangement through the Boron nitride (BN) keeps the discharge within a confined area, and along the magnetic field. This optimizes the exchange interaction between the electrons and vapours.

The advantage of the Danfysik system is that it does ensure



Figure 3: ION GUN ASSEMBLY.

an immediate interaction between electrons and vapours. However a very noticeable disadvantage is that if the gas flow is too great, more neutrals can be pumped down the column before the vapours have a chance to ionize. Therefore, the number of positive ions decrease and the arc will eventually extinguish. The controllability of the gas feed system becomes a very major factor in maintaining the operations of this ion source.

From within the ion source the ions leave the gun assembly through the cathode extraction port, which is maintained at a negative potential. The configuration of the plasma or meniscus at this point is dependent upon the size of port as well as the potential at the extraction electrode.

## Ion Extraction Assembly

In a space in which a considerable number of ions are produced, the resulting potential well would immediately cause the attraction of the slow electrons for space charge neutralization. However some form of a plasma is always formed in the ionization chamber, whether it be the glow discharge or the production of ions. As illustrated in Figure 4, the field about the cathode port and the extraction electrode will be screened from the plasma by the formation of a space-charge sheath. As mentioned previously, at the boundary between the space-charge sheath and the plasma, the electric field is very small (equal to the penetrating field) and positive ions drift through the sheath. The total ion current at the plasma boundary is defined by the area and the current





arc plasma.

density. Figure 4 illustrates how the increase in extraction potential re-shapes the plasma into the initial cylindrical beam of ions.

Let us first assume that the extraction electrode is electrically neutral, then the plasma will extend by diffusion out of the cathode extraction port into the space between the ion gun and the extraction electrode, Figure 4(a). However within this space, the density of positive particles will be much smaller than within the plasma region of the gun. About the extraction electrode, a space charge sheath will be formed whose characteristics are given in accordance with Langmuir's theory for space-charge-limited currents<sup>(3)</sup>.

Now if a negative voltage is applied to the extraction electrode, the plasma will be pushed towards the extraction aperture while the plasma boundary takes on the shape of a so-called meniscus with area (Am). As the extraction voltage increases, the meniscus will tend to bulge more towards the plasma. Providing the meniscus does not extend too far into the plasma, the current  $I_+$  of the beam towards the extraction electrode will remain constant; it being equal to the ion-diffusion current  $J_+$  through the aperture area, Am.

Variations to the extraction voltage will influence the beam shape for two reasons: firstly, by changing the meniscus configurations; and secondly, by deforming the boundaries of the plasma within the spacecharge region. Consequently, this portion of the ion source is uttermost in defining the geometry of the beam before it proceeds any further down the column.

# PART II BEAM TRANSPORT SYSTEM

The present system, illustrated in Figure 5, consists of an ion source, einzel lens, accelerating column, an analyzing magnet, and a series of apertures and deflection plates. The electronic system for the deflection plates has not as yet been constructed. However, when the deflection system becomes operative it will be used to sweep the ion beam uniformly across the sample surface. A thorough study of the beam transport system permits the mathematical presentation of those parameters crucial for obtaining a well-focussed beam (stigmatic: beam). Any changes that will inevitably have to be made to these parameters, as future researchers dictate, can then be predetermined. This, in turn, can save the researcher invaluable time as he or she will then be able to utilize the equipment about the needs of the experiment instead of visa versa.

A study of the beam transport should begin at the point where the ions exit from the cathode extraction port. However, if the distance between the gun assembly and the extraction electrode is fixed and, providing the ion beam can move freely through the extraction ports without any angular obtrusions, then the object size of the beam can be approximated as the size of the extraction electrode port, Figure 6(a). From this point, down and through the analyzing magnet can be treated graphically.



Figure 5: Schematic of the Beam Transport System.



Figure 6(a): Ray trace illustrating the use of an Einzel Lens to form a stigmatic image.



Figure 6(b): Dimensional schematic of the 150 kV ion source.

## Focusing and Accelerating the Ion Beam

Focusing of the emerging ion beam is performed by the einzel lens (Figures 6(a) and 6(b)). The einzel lens is treated as a compounded Double-Aperture lens of the type described by Zworykin<sup>(5)</sup> for which the mathematical derivation is given in Appendix I. Figure 7 is a plot of the lens focal length (cm) against the voltage ratio (Q) between the lens plate. From Figure 6(b), Figure 7 and Table III, it can be appreciated that the optimum operating conditions for the lens are with the voltage ratio between 2.5 and 3.5. This permits the focal point to be within the portion of the lens column such that the image is a well-defined area of about 2 mm in diameter before accelerating any further down the line. Decreasing Q weakens the electric field <sup>4</sup> about the accelerating plates of the lenses and thereby increasing the focal length towards the accelerating column. This, in turn, increases the area of the image being presented to the accelerating column which spreads the same current density over a greater target area.

Galejs and Rose<sup>(6)</sup> treat the accelerator column as a Double-Aperture lens as described by Equation (A1-12) in Appendix I. Even though the topic in itself is more complex mathematically than this, the technique proved to be quite adequate providing certain reasonable assumptions were made. It was assumed that the potential distributions for the entrance and exits to the column were mirror images and, that the middle of the column was represented by a uniform field with a potential distribution  $V_0$  along the total length of the column.

Using the above technique, trajectories for the 150 kV were



<u>Figure 7</u>: Einzel Lens 5 kV Extraction ( $V_1$ ); d = 7 cm.

	and all second se	* 					
Q	V <sub>2</sub> (kV)	<sup>a</sup> 21	f <sub>f</sub> (cm)	<sup>a</sup> 11	1 <sub>0</sub> (cm)	] <sub>i</sub> (cm)	1 <sub>0</sub>   +  1 <sub>1</sub>  (cm)
1.5	7.5	0.64	109.03	0.911	9.70	-10.64	20.34
2.0	10.0	1.80	38.80	0.785	8.34	-10.62	18.96
2.5	12.5	3.10	22.60	0.677	7.30	-10.78	18.08
3.0	15.0	4.23	16.54	0.532	7.74	-14.55	22.29
3.5	17.5	4.94	14.18	0.393	8.61	-21.90	30.51
4.0	20.0	5.62	12.45	0.250	9.34	-37.35	46.69
4.5	22.5	6.06	11.55	0.108	10.30	-45.39	105.69

TABLE II: EINZEL LENS SETTING (V2) d = 7 cm Lens 5 kV Extraction ( $V_1$ )

TABLE III: ACCELERATOR SETTING  $(V_f)$ d = 47 cm5 kV Extraction  $(V_1)$ 

Q	V <sub>f</sub> (kV)	<sup>a</sup> 21	f <sub>f</sub> (cm)
2.0	10.0	0.1652	605.16
2.5	12.5	0.3162	354.00
3.0	15.0	0.4769	256.81
4.0	20.0	0.8462	167.11
5.0	25.0	1.2477	126.74
10.0	50.0	3.4719	64.40
15.0	75.0	5.8591	46.74
20.0	100.0	8.3225	37.99
25.0	125.0	10.8322	32.63
30.0	150.0	13.3741	28.959

calculated in the composite accelerating field for the ratio of output to input beam energies (Q) from  $5 \le Q \le 30$ , Table IV.

Referring to Figures 7 and 8 as well as Figure 6(b), the question can now be answered as to the best possible position for locating the einzel lens. The position which will accomodate movement of the required object position for the greatest range of accelerating potentials. Figure 8 fixes the centre of the lens at approximately 15 cm from the extraction port. This will allow the distance  $(1_0)$  to vary over a 10 cm range for a change in focal length  $(f_f)$  between 22.6 to 14 cm (Table III) and still maintain a 2 mm object for acceleration. This calculated location for the einzel lens agrees with the "new position" recommended by the manufacturer of the ion source.

The new location of the lens should not only improve focusing but will help to eliminate flashovers between  $V_2$  to  $V_1$  (Appendix I, Figure A1-6). This occurs due to the glow discharge, which was previously mentioned, existing in the vicinity of the extraction electrode. Another alternative to limiting the flashover is to increase the pathlength between the electrodes by increasing the outer diameter of the insulator spacers between the lens plates. This can be readily performed without changing the value of "d", the distance between the plates.

Figure 9 shows the relation between Q and the expected focal lengths for the present accelerating column. Once the einzel lens is operating properly, Figure 9 can be used to determine the best possible location for a beam current indicator (beam stop). The proper location







d = 47 cm.

for the beam stop is important for start-up so that the ion source parameters can be peaked for the optimum operating efficiency (Appendix III).

## Mass Separator

Mass separation for the present system is performed by an analyzer magnet, Figure 10. The centre of an ion beam of momentum  $(p_0)$  can be made to follow a circular radius  $(\rho)$  and angle  $(\psi)$  through the influence of a magnetic flux (B), related by the equations:

$$\frac{1}{\rho} = \frac{e^{B}o}{\rho_{0}} = \frac{0.69B \text{ (gauss)}}{\sqrt{M} \sqrt{V} \text{ (volts)}} \text{ (meters}^{-1}\text{)}$$

$$\psi = \ell/\rho , \qquad (7)$$

where *L* is the length of the ion path in the magnetic field. Figure 11 is a plot of B versus M for an accelerator potential of 80 kV using the existing magnetic separator in conjunction with the 150 kV Danfysik ion source.

Beam transport through the magnet can also be treated using a transfer matrix. In addition to its bending action on a charged particle, which in effect provides the desired mass dispersion, the analyzing magnet can cause a focusing action on those particles in the plane of trajectory. For a more extensive analysis of an analyzing magnet and its focusing action on particles, should the problem become apparent, an excellent review on the subject can be found in either Enge<sup>(7)</sup> or Staffen<sup>(8)</sup>. From Enge<sup>(7)</sup>, the focal lengths about the magnet (Figure 10(b)) for the object (L<sub>1</sub>) and the image (L<sub>2</sub>) are given by Barber's



Figure 10(a): Mass Separation by Analyzing Magnet.



Figure 10(b): Illustrating focusing from an analyzing magnet (Barber's rule: object and image are on a line through the centre of curvature).





rule and are related by,

$$L_2 = L_1 \left[ \frac{1 + (\rho/L_1) \tan \phi}{(L_1/\rho) \tan \phi - 1} \right]$$

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# PART III CRYOGENIC EXPERIMENTAL CHAMBER

The cryogenic experimental chamber (Figure 12) was designed for low-temperature and clean-environment channeling studies. The equipment consists of a cryomite refrigerator (A) coupled to the target chamber (B) by the standard dependex vacuum seal. Attached to the cryomite cold head, inside the vacuum chamber, is a long cylindrical copper block (C). The block is electrically isolated from the rest of the refrigeration system and from ground potential by a saphire crystal (D).

A liquid nitrogen cryoshield (E) is used to reduce radiation <sup>4</sup> heating from the target chamber, from affecting the cooling efficiency to the cylindrical copper block, the target holder (F), and the hemispherical cryoshield (G). The copper hemisphere is attached at (H) to the cylindrical copper block. Its main purpose is to act as a cryoshield for the sample surface. There are sections where the sample surface faces areas of higher temperature. These are through the openings directed towards the solid state counter (I) and the beam line. The solid state counter is a press fit mount onto the liquid nitrogen shield.

The target holder is thermally connected to the cylindrical copper block by a 10 cm length of copper braid (J). The braid is approximately 0.6 cm in diameter, consisting of either numbers 32 to 40 gauge wires wrapped tightly together. The braid was then wrapped in teflon tape to ensure the strands stayed together and to prevent



Figure 12: Cryogenic Experimental Chamber (see Table IV for parts listing).

# TABLE IV

- A cryomite
- B target chamber (experimental chamber)
- C cylindrical copper block
- D saphire crystal (insulator)
- E liquid nitrogen shield
- F target holder
- G hemispherical cryoshield
- H attachment of hemispherical cryoshield to cylindrical copper block
- I solid state counter (detector)
- J copper braid
- K 15 watt heater
- L thermocouple (chromal-gold (2% iron))
- M stainless steel tubing, target holder support.

unravelling during rotation of the target hold. Rotation to the target holder is performed by a standard 2-axis goniometer assembly.

A 15 watt heater (K) for the target holder is supplied by two diodes wired in series. Supplemental heating can be supplied through the control system for the cryomite. Using these two heating systems, singularly or in tandem, will provide a controllable temperature ranging between 40-300°K. Temperature recording is done with a thermocouple made of chromal and gold (2% iron). The thermocouple (L) is attached to the target holder in close proximity to both the heater supply and where the sample is fastened. To minimize thermal gradients affecting the thermocouple measurements, the leads for the thermocouple were brought down from the copper target holder and passed through the centre of the stainless steel target holder support tubing (M).

A 36 cm long piece of coaxial cable was used to make the connection between the solid state counter and the preamplifier located on the outside of the experimental chamber. This was found to reduce the occurrance of microphonic pick-up when the cryomite was turned on. The grounding shield of the coaxial cable was attached to ground at the vacuum feed-through end and was left floating at the solid state counter connection.

### Start-Up

Before closing the vacuum chamber to atmosphere, several precautions should be taken in order to eliminate damaging the equipment. Firstly, all connections should be checked for continuity or possible

short circuits. No interconnections should exist between the thermocouples, heater supply and especially the beam current lead. It is the beam current lead which made it necessary to electrically isolate the cryomite cylindrical copper block from ground. Secondly, precautions should be taken to insure that the sample holder can rotate through the required number of revolutions for the experiment. Otherwise, the electrical leads may become entangled and, in doing so, become damaged.

After the pumpdown is completed and the experimental chamber has been put onto the diffusion pump, then the trap for the liquid nitrogen shield can be filled. The overall time required to bring the sample holder to equilibrium (77°K) is about one hour. At this point, the cryomite can be turned on, making sure at first that the cryomite temperature controller is either off or set to zero. Figure 13, temperature versus time for the cryomite, shows that it takes between 15 to 20 minutes before the sample holder reaches the minimum value of 40°K.

Notes of Caution: 1) The gate value isolating the experimental chamber from the rest of the beam line should not be opened during low temperature measurements until the cryoshields and target holder have reached the minimum temperature settings. Otherwise, carbon or other contaminance build-up will occur on the sample surface. For the present system, if operated as prescribed, it was not possible to detect any build-up of impurities during a 24 hour experimental period regardless of the target temperature.

2) The solid state counter (detector) should not be powered



Figure 13: Temperature versus Time to cool target holder.

while the experimental chamber is at atmosphere and should only be turned on after the chamber goes on the diffusion pump. Similarly, the detector, and therefore the liquid nitrogen cryoshield, should not be brought up to ambient temperature too quickly or by using a "hot" air gun. The best possible procedure is to use the air gun to blow room temperature air into the liquid nitrogen filling trap. It will take approximately one to two hours to bring the inside shield and detector up to room temperature, but no damage will be done to the solid state detector.

## DISCUSSION

This report is not intended as an operating or repair manual for the 150 kV accelerator and related cryogenic experimental chamber. Rather, its function is to illustrate the main constituents of the overall system, emphasizing the most important items, in order that the system works properly!

It is with deep regret, however, that the writer has not been able to proceed with the intended research for which this system was initially constructed. The problem stems from the Danfysik ion source. As a piece of machinery, it is a white elephant; as a design concept, it appears to be quite good judging from an evaluation of its dimensional parameters and operating characteristics: It operated successfully for about 6 hours. Where it lacks, however, is in the workmanship, the quality of materials used in the construction, and the amount of information supplied by the company for setting up and operating the source.

According to High Voltage Engineering, the North American representative for Danfysik, all ion sources are pretested before they leave Denmark. However, in our first run, the extraction electrodes shorted together under vacuum, damaging the extraction voltage supply. Later, it was discovered that the extraction port had not been machined to tolerance and was sitting at an angle in its positioning flange. Both the extraction electrodes and port had to be re-machined before an

ion beam could be extracted from the gun. Before the faults could be found and rectified, the angled ion beam had apparently been hitting the plastic einzel lens electrode, managing to distort the "o"-ring seal.

Separating the extraction electrodes is a 12.5 inch annularshaped plastic insulator. This insulating piece is also used to keep the alignment between the ion gun assembly and the extraction electrode port, which as illustrated in this report is crucial for obtaining a well-formed (stigmatic) beam. Even though the whole ion gun assembly requires no more than 200 to 230 watts, there is sufficient heat generated to cause the piece of plastic to soften. This softening, not only mis-aligns the ion beam, but has been the cause of vacuum breaks on several occasions.

Without going any further into a long elaboration, the following list describes the corrections now being made, and found necessary before the "Danfysik" ion source can operate successfully:

 Remaking the annular disc; - material Kel-F thermal plastic; softening temperature 200°C.

Install a water-cooled heat exchanger; - deionized water for
 150 kV with corona shield in place. This should eliminate any possi bility of beam mis-alignment due to overheating.

3) Redesign the einzel lens system; - at present, (i) the lens is not located for proper focusing of the ion beam, (ii) increasing the diameters of the lens mounting insulators to eliminate flashovers between  $V_2$  and  $V_1$ . Described in the section of this report on Beam Transport, Part II, the new location for the einzel has been calculated. The new position will now allow the beam to be focussed over a range of accelerating voltage and independent of the accelerator potential. Previously, it was the accelerator voltage, set at 50 kV, which had been doing the focusing. Consequently, unless a beam with an energy of 50 kV was desired, most of the ion current never reaches the target area.

4) The redesign of the gas flow system; this meant drilling out a section of the boron nitride oven, Figure 3, and installing a piece of the same material that would make a proper fitting between the gas input tube and the ionizing chamber. The redesign of this means that the oven assemblies as supplied by Danfysik are of no value.

5) Redesign the oven assemblies - fortunately, these have not been required for any initial runs, therefore, nothing has been done.

6) Boron nitride (BN) is contaminated by organic compounds; any slight amount of back streaming from an oil diffusion or mechanical pump will leave some trace of oil on the BN insulators. Thus, oil film build-up appears to increase when the ion source is shut down for a few days. The build-up of oil decomposes to form a carbon conducting path between the cathode and anode electrodes, eventually shortening out and causing the ion discharge to extinguish.

7) Stainless steel screws and threads used to support the ion gun assembly either "corrode" or "deform" (the mechanism is not yet fully understood) such that it becomes impossible to disassemble the gun. Correction for this seems to be a thin coating of molybdenum

disulphide (MoS<sub>2</sub>) on all stainless steel fasteners used on the gun assembly.

The seven items above are the most pressing problems before the 150 kV ion source is ready for operation. Although the theory appears to be good, the workmanship, materials used, and the cooperation of the manufacturer leaves an awful lot to be desired!

### APPENDIX I

Kirstein<sup>(9)</sup> gives the equation for paraxial ray equations in an axially symmetric electrostatic field only as

$$\frac{d^2r}{dz^2} + \frac{V_0'}{2V_0}\frac{dv}{dz} + \frac{V_0''r}{4V_0} = -\frac{\rho r}{4V_0\varepsilon_0}, \qquad (AI-1)$$

where  $V_0(z)$  is the potential in the axis and the primes denote derivative with respect to z; r is the radial component  $(x^2 + y^2 = r^2)$  of the ray;  $\rho$  represents the space-charge density assumed uniform over the beam cross section; and  $V_0^{"}$  relates to the focusing as less action of ' the structure.

Assuming that the space-charge density is negligible, then the right hand side of Equation (AI-1) is equal to zero. This is then a linear second-order homogeneous differential equation with coefficient depending upon z. Boundary conditions for solutions to this equation are normally set between two planes located a given distance apart along the axis. Therefore, if solutions can be found for the ray path within these boundaries, then any other ray path can be expressed in terms of these solutions.



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Boundary conditions for these special ray solutions:

$$r_{\alpha_0} = 0; r_{\alpha_0} = 1; r_{\beta_0} = 1; r_{\beta_0} = 0$$

where subscript 0 refers to the object plane and i to the image.

Solution to the paraxial ray equation (AI-1) is given (10) as

$$r_{i} = r_{\beta}r_{0} + r_{\alpha}r_{0}',$$

$$r_{i}' = r_{\beta}r_{0} + r_{\alpha}r_{0}'.$$
(AI-2)

Coefficient  $a_{\mbox{ij}}$  can be related to the parameter of the special  $\alpha$  and  $\beta$  rays by

$$a_{11} = r\beta_i; a_{12} = r\alpha_i; a_{21} = r'\beta_i; a_{22} = r'\alpha_i$$

$$\begin{pmatrix} r_{i} \\ r_{i}' \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{pmatrix} \begin{pmatrix} r_{o} \\ r_{o}' \\ r_{o}' \end{pmatrix}$$
(AI-3)

The relationship between the matrix element and the cardinal points (Figure AI-2) are given as <sup>(6)</sup>;

$$M_{16} = \begin{vmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{vmatrix}$$

$$f_i = \frac{-\sqrt{V_i}}{a_{21}}$$
, object - plane focal length (AI-4)

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Figure AI-1(b): Einzel Lens

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d = length of lens.

Figure AI-2: Definition of Cardinal Points.

$$f_f = \frac{\sqrt{V_f}}{a_{21}}$$
, image - plane focal length (AI-5)

$$z_{fi} = \sqrt{V_i} \frac{a_{22}}{a_{21}}$$
, location of object - plane focal point (AI-6)

$$z_{ff} = d - \sqrt{V_f} \frac{a_{11}}{a_{21}}$$
, location of image - plane focal point (AI-7)

 $z_{ni} = z_{fi} - f_i$ , location of object - principal plane (AI-8)

$$z_{hf} = z_{ff} - f_{f}$$
, location of image - principal plane (AI-9)  
where d is the overall length of the lens assembly.

For stigmatic conditions the relationship between the object  $(1_0)$  and the image  $(1_i)$  distances are:

$$a_{12} = l_i (1 - \frac{l_o}{f}) + l_o = 0$$
 (AI-10)

and the magnification factor is

$$a_{11} = 1 - \frac{1_0}{f}$$
 (AI-11)

# Double-Aperture Lens

The Double-Aperture Lens as derived by Zworykin<sup>(5)</sup> asserts that the lens beam transfer matrix can be formed by multiplying together the appropriate matrices, for the initial and final breaking points in the electric field together with the uniform field region (refer to Figure AI-1(a)). The matrix elements referred to are:

$$M_{12} = \begin{vmatrix} 1 & 0 \\ \frac{V_1 - V_2}{4d\sqrt{V_1}} & 1 \end{vmatrix}$$

$$M_{23} = \begin{vmatrix} 1 & & \frac{2d}{\sqrt{V_1} + \sqrt{V_2}} \\ 0 & & 0 \end{vmatrix}$$

$$M_{34} = \begin{vmatrix} 1 & 0 \\ \frac{V_2 - V_1}{4d\sqrt{V_2}} & 1 \end{vmatrix}$$

Combining these matrices, the transfer matrix is;

$$M_{14} = M_{34} \cdot M_{23} \cdot M_{12}$$

$$M_{14} = \begin{pmatrix} \frac{3\sqrt{V_1} - \sqrt{V_2}}{2\sqrt{V_1}} & \frac{2d}{\sqrt{V_1} + \sqrt{V_2}} \\ \frac{(3(V_2 - V_1)}{8d})(\frac{\sqrt{V_1} - \sqrt{V_2}}{\sqrt{V_1V_2}}) & \frac{3\sqrt{V_2} - \sqrt{V_1}}{2\sqrt{V_2}} \end{pmatrix}$$

(AI-12)

An einzel lens can be approximated if we multiply together matrices of the form used for a double-aperture lens. By reversing the notation in the second field region (Figure AI-1(b)), the matrix element becomes

$$M_{16} = (M_{34} \cdot M_{23} \cdot M_{12}) \cdot (M_{34}' \cdot M_{45}' \cdot M_{56}')$$

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$$\frac{\frac{16\sqrt{V_{1}V_{2}}-6V_{1}-6V_{2}}{4\sqrt{V_{1}V_{2}}}}{\frac{2d}{\sqrt{V_{1}}+\sqrt{V_{2}}}\frac{3\sqrt{V_{2}}-\sqrt{V_{1}}}{\sqrt{V_{2}}}}{\frac{3}{\sqrt{V_{2}}}}$$

$$\frac{3}{8}\frac{(V_{1}-V_{2})}{d}(\frac{\sqrt{V_{2}}-\sqrt{V_{1}}}{\sqrt{V_{1}V_{2}}})(\frac{3\sqrt{V_{1}}-\sqrt{V_{2}}}{\sqrt{V_{1}}}-\frac{16\sqrt{V_{1}V_{2}}-6V_{1}-6V_{2}}{4\sqrt{V_{1}V_{2}}}}{4\sqrt{V_{1}V_{2}}}$$
(AI-13)

M<sub>16</sub> =

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# APPENDIX II

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## TABLE OF RECOMMENDED SOURCE FEED COMPOUNDS

Partial Reprint From

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"The Technology and Chemistry of Heavy Ion Sources"

by

J.H. Freeman and G. Sidenius

Z	ELEMENT M E <sub>i</sub> AMU eV	COM- POUND	GAS Com. Sup. Tr.	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	Pressu 10 <sup>-2</sup>	s, <sup>o</sup> c. res, t 760	orr. Nelt.	-1- °C- 1	ION S	OURCE - -3- po -11	TYPES	-S- Surf.	REMARKS
1	llydrogen 1 13.5	H2	Н2	-268	-267	-266	-253	-259	r	r	r	r	i	Explosive in mixture with air.
2	Kelium 4 24.5	Не	Ke	1	1	/	-259	-272	r	r	r	r	i	No protlems.
3	Lithium 7 5.4	Li LíCl	NG	404 458	467 514	537	1317 1380	179 614	i S.p i	p d.	p r	.p r	r P	Require special handling. Also other halides.
4	Beryllium .9 9.3	Be BeCl <sub>2</sub> BeF <sub>2</sub>	NG CC14	990 ./ ./	1097 175 562	1227 209 632	2470 481 1159	1280 405 803	i S.p i i	i C.p p d	i C.r P T	r P r	1	Toxic, ( BeO ). Also BeBr <sub>2</sub> & BeI <sub>2</sub> . More stable temperatur control.
s	Boron 11 8.3	B BC13 BF3	NG CC14 BC13 BF3	1707 7. 1	1867 ./ /	2027	3600 13 -101	2300 -107 -128	i S.p d d	i S.p P P	i C.r P p	r P P	i i	llydrolyse very readily.
6	Carbon 12 11.2	co co <sub>2</sub> cc1 <sub>4</sub>	co co <sub>2</sub> cc1 <sub>4</sub>	-238 -177 /	-235 -167 /	-231 -158 /	-191 -73 77	-207 / -24	r . r	r r P	r r p	r r p	i i i	Toxic, but gives best yield. Most convenient.
7	Nitrogen 14 14.5	N <sub>2</sub>	N <sub>2</sub>	-242	-239	-235	-196	-210	r	r	r	I.	j	Dominating molecular beam.
8	0xygen 16 13.6	0 <sub>2</sub> cc c0 <sub>2</sub>	02 CC CO2	-236 -238 -177	-233 -235 -167	-230 -231 -158	-183 -191 -78	-218 -207 /	P P r	P p r	P P r	Pp r	i i i	Some source and filament cor- Toxic. rosion Gives best yield.
9	Fluorine 19 17.3	r <sub>2</sub> KF	F2	·/ /	/	-231	-188	-220	p i	p i	p r	p r	1	Toxic. Also many other fluorides.
10	Neon 20 21.5	Ne	Ne .	-263	-262	-261	-246	-249	r	r	Г	r	i	No problems.
11	Sodium	Na	NG	193	235	289	880	98	1.	P	P	P	r	Require special handling.

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ELEMENT M E <sub>i</sub> Amu eV	COK- Pound	GAS Com. Sup. Tr.	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	PATURE Preseu 10 <sup>-2</sup>	s, <sup>o</sup> c. res, to 760	orr. Melt	-1- °c- 10	10N 50 -2- 10 - 5	OURCE 1  -3-  00 - 11	eypes -4- 100-°C	-5- Surf.	ackarks .
2 Magnesium 24 7.6	Mg MgCl <sub>2</sub>	NG	327 479	377 532	439 596	1100 <sub>.</sub> ]412	651 708	i S.p i S.p	р J	r P	r P	i <sup>.</sup> 1	Also Mggr, and MgI2.
3 Aluminium 27 6.0	A1 AlCl <sub>3</sub>	NG CC14	97? /	1082	1217 /	2467 183	660 190	i S.p 1	i C.p P	р С.т	r P	p d	Temperature control difficult.
4 Silicon 28 8.1	Si SiCl <sub>u</sub> SiS <sub>2</sub>	NG CCl <sub>4</sub> SiC <sub>4</sub>	1337 / /	1472 /. /	1632 / /	3140 59 1090	1410 -70 Subl.	i S.p p i	i S.p ? d	i C.p r r	r r p	1 1 1	Also SiC <sub>2</sub> . Also Sir <sub>y</sub> .
5 Phosporus 31 10.9	P4 PC13 PH3	NG PC13 PH3.	129 / /	157 <sup>.</sup> /	185	420 75 -88	/ -28 /	d. P P	P P P	r P P	r P P	i i i	Careful handling required. Hydrolyse very readily. Toxic.
5 Sulphur • . 32 10.3	s so <sub>2</sub>	NG SO <sub>2</sub>	55 -145	80 -135.	109 -124	445 -10	115 -75	P r	p r	r	r	i i	Many other sulphur compounds possible.
7 Chlerine 35 13.0	C1 <sub>2</sub> CC1 <sub>4</sub>	C12 CC14	-164	-155 /	-145 /	•35 77	-103 -23	P r	p r	P r	p r	i i	Also a wide variety of other chlorides.
8 Argon 40 15.7	Ar	Ar	-237	-234	-230	-186	-169	r	r	r	r	i	No problems.
9 Potassium 39 4.5	к КС1	NG	123 516	161 572	208 637	770 1500	64 776	i i S.p	r p	r P	r P	r p	Require careful handling. Also other halides.
0 Calsium 40 6.1	Ca CaCl <sub>2</sub> CaO	Ne CCl <sub>4</sub>	459 732 1727	522 814 1875	597 902 2050	1480 1600 2850	842 772 2580	i S.p <u>:</u> i	d i i·	r p i C.r	r p c C.p	P P r	Also other halides. Also CaCO <sub>3</sub> ,decomp. te CaO+CO <sub>2</sub> .
1 Scandium 45 6.7	Sc ScCl <sub>3</sub> Sc <sub>2</sub> 0 <sub>3</sub>	NG CC1 <sub>4</sub> CC1 <sub>4</sub>	1107 662 /	1232 717 /	1377 787 /	3000 / /	1539 939 /	i S.p i i	i S.p d i	d Ċ.p r i C.r	r r d C.p	d d r	Also other halides. Also ScC.

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Z ELEMENT M E <sub>i</sub> Amu ev	COM- Pound	GAS Com. Sup. Tr	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	RATURE Pressu 10 <sup>-2</sup>	s, <sup>o</sup> c. res, t 760	orr. Melt	-1- °C- 1	ION 5 -2- 00 - 5	OURCE - 	TYPUS -4- 00 -°C	-5- Surf.	REMARKS
2? Titanium 48 5.8	Ti TiCl <sub>u</sub> TiO <sub>2</sub>	NG CC1 TiC1 CC1	1442 / 1780	1577 / .1930	1737 / 2100	3260 136 2750	1675 -30 1840	i S.p d i	l S.p d i	I C.p p i C.r	r p d C.p	d d r	As the lanthanides. Also Ti <sub>2</sub> 0 <sub>3</sub> .
23 Vanadium 51 6.7	V VF <sub>3</sub> VO <sub>2</sub>	אק ככו <sub>4</sub> ככו <sub>4</sub>	1547 / /	1687 687 /	1847 765 /	3360 / /	1830 / 1967	i S.p i i	i S.p i i	i C.p r i C.r	r p, d C.p	d d r	Also other halides. Also other oxides.
24 Chromiun 52° 5.7	Cr CrCl <sub>3</sub> Cr <sub>2</sub> 0 <sub>3</sub>	NG CC14 CC14	1157 /	1267 509 /	1397 / /	2650 949 /	1890 1150 1990	i S.p i i	i S.p d i	d C.p r i C.r	r p d C.p	d d r	Also other halides. Also other oxides.
25 Manganese 55 7.4	Nn MnCl <sub>2</sub> MnO	NG CC14 CC14	747 / 1349	837 / 1485	937 / 1628	2037 1231 /	1744 650 1705	i S.p i i	i S.p i i	p C.r p i C.r	r p d C.p	i i i	Also other halides. Also other oxides.
25 Iron 55 7.8	Fe FeCl <sub>2</sub> FeO	NG CCl <sub>4</sub> CCl <sub>4</sub>	1227 / 1371	1342 428 1500	1477 492 1640	2860 1012 /	1535 677 1420	i S.p i i	i S.p d i	i C.p r i C.r	г Р р С.р	i i	Also other halides. Also other oxides.
27 Cobalt 59 7.8	Co CoCl <sub>2</sub> CoO	אט געז <sup>ת</sup> ככו <sup>4</sup>	1257 507 1297	1382 617 1410	1517 672 1550	2870 1053 /	1492 740 1800	i S.p i i	i S.p i i	i C.p r i C.r	r P p C.p	i i	Also other halides. Also other oxider.
28 Nickel 58. 7.6	NÍ NIC12 NIO	NG CC14 CC14	1262 / 1240	1382 491 1356	1527 545 1485	2890 973 /	1453 1001 1990	i S.p i i	i S.p d i	i C.p r i C.r	r p p C.p	i i	Also other halides. Also Ni <sub>2</sub> 0 <sub>3</sub> .
29 Copper 63 7.7	Cu Cu <sub>2</sub> Cl <sub>2</sub> Cu0	NG CCl <sub>4</sub> CCl <sub>4</sub>	1027 / /	1137 / 925	1257 / 1070	2580 1366 /	1083 422 1326	i S.p i i	i S.p i i	p C.p p p C.r	r P P	i i i	Also CuCl <sub>2</sub> , which decomposes to Cu <sub>2</sub> Cl <sub>2</sub> .
30 Zinc 64 9.4	Zn ZnS	KG	247 779	297 863	3411 962	9 <u>0</u> 7 1185	417 sut1.	i S.p i ·	r 1	r P	r P	i	No particular problems.

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Z ELEMENT H E <sub>1</sub>	COM- POUND	GAS Com.	For	TEMPE Vapor	RATURE Pressu	s, <sup>o</sup> c. res, t	orr.	-1-	ION 50	URCE 1	TYPES	-5- SumF	REMARKS
AMU EV		Sup. Tr.	10	10 -	10	1760	Melt	C- 10	10 - 50	-11	- C	SUPI.	
31 Gallium 59 6.0	Ga GaI <sub>3</sub> Ga <sub>2</sub> 0 <sub>3</sub>	NG GaI <sub>3</sub> CCl <sub>4</sub>	907 1 1	1007	1132 /	2400 345 /	30 ?12 1900	i P i	i P i	P r i C.r	r p d C.p	r d r	No partícular problems.
32 Germanium 74 8.1	Ge GeI <sub>4</sub> GcS <sub>2</sub>	NG CC1 <sub>4</sub> GeI <sub>4</sub>	1137 / 406 -	1257 41. 447	1397 65 494	2830 377 600	937 146 800	i S.p p i	i S.p r p	i C.p r r	г Р	i i i	Cl <sup>‡</sup> may cause contamination.
33 Arsenic 75° 10.5	As AsCl <sub>3</sub> GaAs	NG AsC1 <sub>3</sub>	204 /	237 / /	277 / /	700 130 /	810 -18 1238	i p i S.p	p r d	p p r	P P T	i i i .	Toxic. Decomposes controllably in oven.
34 Sclenium 80 9.7	Se ScO <sub>2</sub> CdSo	NG .	164 75 /	199 90 588	243 124 661	680 337 /	217 345 1350	i S.p d i	p r d	r r	r p r	i i i	Toxic. Convenient temperature control.
35 Bromine 79 11.8	Br <sub>2</sub> NaEr	Br <sub>2</sub>	-110 657	-99 751	-84	59 1390	-7 755	r i	r i	r	r	i i	Also other bromides.
36 Krypton 84 13.9	Kr	Kr	-224	-219	-214	-153	-157	r	r	r	r	i	No problems.
37 Rubidium 85 4.2	Rb RbCl Rb <sub>2</sub> CO <sub>3</sub>	NG	94 487 /	129 544 /	173 614 /	700 1380 740	39 715 ∙ 837	i S.p i i	r . P i	r r r	r r p	r P r	Also other halides.
38 Strontium 88 5.7	Sr SrCl <sub>2</sub> SrC0 <sub>3</sub>	NG CC14	404 716 /	465 799 /	537 · 888 /	1370 2000 1340	769 873 1497	i S.p i i	r i i	r p i C.p	г ' р р С.р	r P r	Also other halides.
39 Yttrlum 89 6.5	Y YC] <sub>3</sub> Y <sub>2</sub> 0 <sub>3</sub>	NG CC14 CC14	1332 / 2098	1467 613 7750	1632 698 2412	3300	1495 680 2410	i S.p i i	i S.p i	j C.p p i C.r	r P d C.p	P d r	As the lanthanides.

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7 ELEMENT H E <sub>i</sub> Amu ev	COM- POUND	CAS Com. Sup. Tr.	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	RATURE Pressu 10 <sup>-2</sup>	S, <sup>O</sup> C. res, t ¦760	orr.  Melt	-1- °C- 1	ION S -2- 00 - 5	OURCE 1 1-3- 00 -110	rypes  -4- 00 -°c	-5- Surf.	Remarks
40 Zirconium 90 6.9	Zr ZrCl <sub>y</sub> ZrO <sub>2</sub>	KG CCl <sub>4</sub> CCl <sub>4</sub>	1987 82 2077	2177 103 2240	2397 128 2400	4500 331 5000	1852 437 2677	i S.p i i	i S.p d i	i C.p p i C.r	p p d C.r	d d · P	As the lanthanides.
41 Niobium • 93 6.9	ND ND2 <sup>0</sup> 3	NG CC14 CC14	2277	2447	2657	4400	2468	i S.p i	i S.p i	i S.p i C.r	p d C.r	d P	As the lanthunides.
42 Molybdenum 98 7.4	Мо Мо0 <sub>3</sub>	NG ĊC1 <sub>4</sub>	2117	2307	2527 646	4620 1150	26]0 795	i S.p i	i S.p i	i S.p p C.r	р рС.г	i	Also MoO2
43 Technetium (99) (7.5)	Tc TcO <sub>x</sub>	ccių	2077 /	2257	2487	4580	2200	i i	i	i i C.p	p d C.p	i	No stable isotopes.
44 Ruthenium 102 7.7	Ru RuF <sub>5</sub>	NG RuF <sub>5</sub>	1987 /	2147 31	2347 50	4070 270	2250 106	i S.p i	i S.p i	i S.p	r	i i	Only very unstable compounds.
45 Rhodium 103 7.7	Rh	NG	1707	1255	2037	3700	1960	i S.p	i S.p	i S.p	r	i	Only very unstable compounds.
46 Palladium 106 8.3	Pd	NG	1192	1317	1462	3020	1550	í S.p	í S.p	d S.p	r	i	Only very unstable compounds.
47 Silver 107 7.5	Ag AgCl	NG	823 539	927 607	1027 685	2212 1560	961 455	i S.p i	i S.p i	r r	r P	i i ·	No particular problems.
48 Cadmium 114 9.0	C92 C9	NG	177 /	217 610	265. 686	767 1382	321 1750	i S.p i	r 1	r r	r r	i i	No partícular problems.
49 Indium 115 5.8	In InCl <sub>3</sub>	NC CC14	742	837	947 256	2100 470	156 586	i S.p i	i C.p r	r P	r P	r P	No particular problems.
50 Tin 120 7.3	Sn SnCl <sub>4</sub>	NG CC14 SnC14	997 /	1104	1747	2630 120	232	i S.p d	i C.p	d C.r P	r P	i i	Also with H <sub>2</sub> S as transport gas.
51 Antimony 171 8.5	Sb	NG	475	475	535	1650	631	i S.p	р	r	r	1	Toxic, no problems.

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Z ELEMENT M E <sub>i</sub> AMU eV	COM- POUND	GAS Com. Sup. Tr.	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	RATURE Pressu 10 <sup>-2</sup>	S, <sup>o</sup> C. rcs, t 1760	orr.  Melt	-1- °C- 10	ION SC -2- }	)URCE 1  -3- 00 -110	TYPES	-5- Surf.	REMARKS
52 Tellurium 130 9.0	Tc	NG	280	323	374	1000	450	i S.p	P	r	r	i	Toxic, no particular problems.
53 Iodine 127 10.5	r <sub>2</sub> KI AgI	I2	-47 448 722	-30 499 845	-11 564 995	183 1323 /	114 682 /	r i i	r d i	r P P	r p p	i i i	Numerous other iodides. Decomposes controllably.
54 Xenon 132 12.1	Xe	Xc	-205	-199	-191	-107	-11?	r	r	r	r	1	No problems.
.55 Cesium 133 3.9	Cs CsCl	NG	78 448	114 505	155	680 1301	29 642	P i	r i	r	r	r P	Careful handling. Also other halides.
55 Barium 138 5.2	Ba BaCl <sub>2</sub> BaO	NG. CC14	462 734 1297	527 813 1421	610 899 1549	1590 1560 2000	725 962 1923	i S.p i i	d S.p i i	r r i C.r	r P P	r p r	Toxic. Also other halides.
57 Lanthanum 139 5.6	La LaCl <sub>3</sub> La <sub>2</sub> 0 <sub>3</sub>	NG CC14	1422 / 1816	1562 751 1966	1727 825 2176	3330 / 4200	9.20 860 2315	i S.p i i	i S.p i i	i S.p p i C.r	r p d C.r	p d r	Even that volatile halides exist for all lanthanide clements are
58 Cerium 140 6.5	Ce CeO <sub>2</sub>	NC CC14	1377	1522	1697	3468	795	i S.p i	i S.p i	i S.p i C.r	r d C.r	p r	they very hygroscopic and dif- ficult to use. It is recommended to use freshly
59 Praseodymium 141 5.8	Pr Pr <sub>2</sub> 0 <sub>3</sub>	NG CC14	1147	1277	1427 /	3030 1	935 /	i S.p i	i S.p i	i S.p i C.r	r d C.r	р г	prepared oxides to obtain max. reactivity with the CCl <sub>4</sub> .
60 Neodynium 142 5.3	Nd Nd203.	NG CC14	1047 /	1167 <sub>.</sub> /	1307 /	3080	1024 1900	i S.p	i S.p i	i S.p i C.r	r d C.r	P r	
61 Promethium (145) /	Pm Pm <sub>2</sub> 0 <sub>3</sub>	NG CC14	1	1	1	2730	1035	i	i i	d i C.r	r d C.r	p r	No stable isotopes.
62 Samarium 152 6.6	5m 5m2 <sup>0</sup> 3	NG CC14	580	653 /	742	1800	1072	i S.p i	i S.p i	p i C.r	r p C.r	P r	

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Z ELEMENT H E <sub>i</sub> AMU eV	COM- POUND	GAS Com. Sup. T .	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	RATURE Pressu 10 <sup>-2</sup>	s, <sup>o</sup> c. res, te 1760	orr. Kelt.	-1- °C- 10	ION SO 	URCE 1 -3- 00 -110	TYPES -4- 00 -°C	-5- Surf.	REMARY.S
63 Europium 153 5.6	Eu Eu <sub>2</sub> 0 <sub>3</sub>	NG CC14	460 /	532	611 /	1500 /	826	1 S.p 1	d S.p	P d C.r	r p C.r	p r	Even that volatile helides exist for all lanthanide elements are
64 GadoJinium 158 6.7	Gd Cd <sub>2</sub> 0 <sub>3</sub>	NG CC14	1077	1197	1327	2730 /	1312 /	j S.p i	i S.p i	i S.p i C.r	r d C.r	P. r	ficult to use. It is recommended to use freshly
65 Terbium 159 6.7	ть ть <sub>2</sub> 0 <sub>3</sub>	NG CC14	1147	1277	1427	3020 /	1356	i S.p i	i S.p i	i S.p i C.r	r d C.r	P	prepared oxides to obtain max. reactivity with the CCl <sub>y</sub> .
66 Dysprosium 164 8.8	Dy Dy <sub>2</sub> 03	NG CC14	897	997 /	1117 /	2420	1407 2340	i S.p	i S.p	d i C.r	r d C.r	p r	
67 Holmium 165 6.0	но но <sub>2</sub> 03	NG CC1 <sub>4</sub>	947 /	1052	1117 ./	2580 1500	1461 718	i S.p i	i S.p i	i S.p i C.r	r p C.r	P	
68 Erbium 166 6.1	Er Er2 <sup>0</sup> 3	NG CC14	947	105? /	1177	2580 /	1497 /	i S.p i	i S.p	i S.p i C.r	r d C.r	p	
69 Thulium 169 6.2	Tm <sup>Tm</sup> 2 <sup>0</sup> 3	NG CC14	68C /	757 /	847	1750 /	1545 /	i S.p i	i S.p	p i C.r	r d C.r	p r	
70 Ytterbium 174 7.1	Yb Yb <sub>2</sub> 0 <sub>3</sub>	NG CCl <sub>4</sub>	417	482 /	557 :/	1540 /	824	i S.p i	i S.p i	p i C.r	r d C.r	d	
71 Lutetium 175 6.2	Lu Lu <sub>2</sub> 03	NG CC14	1277 /	1412 /	1572 /	3020 /	1652 /	i S.p i	i S.p i	i S.p i C.r	r d C.r	р г	
72 Hafnium 180 6.8	hf Hf0 <sub>2</sub>	NG CC1 <sub>4</sub>	1997 2297	2177 2475	2397 <sup>.</sup> 2678	4430 5400	1652	i Š.p i	i S.p i	i S.p i C.r	p d C.r	d	As the lanthanides.
73 Tantalum 181 7.9	Ta Ta <sub>2</sub> 05	NG CC14	2587	7800 /	3050 /	5230 /	3000 1800	i S.p i	i S.p i	i S.p i C.r	i S.p d C.r	i i	As the lanthanides.
74 Tungsten 184 8.1	w %0 <sub>3</sub>	NG CC14	2760 1079	2980 1136	3230 1176	5530 /	3350 1473	i S.p	i S.p	i S.p i C.p	i S.p p C.p	i	Most compounds unstable.

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Z ELEMENT H AMU	E. cV	COM- POUND	GAS Com. Sup. Tr.	For 10 <sup>-4</sup>	TEMPE Vapor 10 <sup>-3</sup>	RATURF: Pressur 10 <sup>-2</sup>	5, <sup>o</sup> C. res, to 760	orr. Melt.	-1- °C- 10	ION SC -2- 00 - 50	URCE T	YPES	-5- Surf.	REMARKS
75 Rhenium 187	7.9	Re Re <sub>2</sub> 0,	OţCC14 NG	2587 /	2807 133	3067 157	5627 362	3180 subl.	i S.p i	i S.p d	i C.r P	i C.p P	i i	Most compounds very unstable.
76 Osmium 192	8.7	0s 0s0 <sub>4</sub>	0±CC14 0504	2487 /	2687	2917 -52	5300 130	3000 40	i S.p i	i S.p d	i C.r P	i C.p P	i	Toxic. Most compcunds very unstable.
77 Iridium 193	9.1	Ir	NG	2107	2287	2497	4530	2443	i S.p	i S.p	i S.p	r	4	Only very unstable compounds.
78 Platium 195	8.9	Pt	NG	1,747	1907	2097	3827	1769	i S.p	i S.p	i S.p	r	i	Only very unstable compounds.
79 Gold 197	9.7	Au	ng	1132	1252	1397	2770	1063	i \$.p	i S.p	d S.p	ŗ		Only very unstable compounds.
80 Mercury 202	10.4	Hg	NC	-7	16	43	357	-39	r	r	r	r	1	Toxic,no problems.
81 Thallium 205	6.1	T] T1F	NG	463 /	530 81	609 106°	1457 655	304 327	i S.p d	d r	r	r P	r	Toxic,no problems.
82 Lead 208	7.4	РЬ РЬС1 <sub>2</sub>	.NG	547 327	625 385	715 431	1744 954	327 501	i S.p	i S.p P	r P	r P	1'	No particular problems.
83 Bismuth 209	8.0	Bi BiCl <sub>3</sub>	NG	517 /	587 118	672 145	1570 447	271	i S.p	d S.p	r	r P	i i	No particular problems.
84 Polonium (209)	8.4	Po PoCl <sub>u</sub>	NG	221	264	315 7	962 390	254 300	i d	r P	r p	r p	1	No stable isotopes.
85 Astatine (210)	9.5	At	NG	43	65	91.	330	300	P	r	r	r	i	No stable isotopes.
86 Radon (222)	10.7	Rn	Rn	1		1	-62	-71	r	r	r	r	2 3 3 5 5 6 9 8	No stable isctopes.

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Z ELEMENT M E <sub>i</sub> AMU eV	COM- POUND	GAS Com. Sup. Tr.	For 10 <sup>-4</sup>	TEMPE Vapor 110 <sup>-3</sup>	RATURE Pressu 10 <sup>-2</sup>	s, °C. res, t 1760	orr.  Melt	-1- °C- 1	ION SO 1-2- 00 - 50	OURCE 1  -J- 00 -110	-4- 10 -°C	-5- Surf.	REMARKS
87 Francium (223) 4.0	Fr	NG	61	95	137	680	17	đ	r	r	r	r	No stable isotopes.
88 Radium (226) 5.3	Ra	NG	41.7	482	557	1530	700	i	d	r	r	r	No stable isotopes.
89 Actinium (227) 5.8	Ac AcCl <sub>3</sub> Ac <sub>2</sub> 0 <sub>3</sub>	NG CC1,	1332 / /	1467 / /	1632 / /	3200 960 /	1050 / /	i i i	i P i	i p i C.r	r P d C.p	d d r	No stable isotopes. As the lanthanides.
90 Thorium 232 7.0	Th ThCl <sub>4</sub> ThO <sub>2</sub>	NG CC1	1997 / 2080	2167 / 2240	2407 /_ 2420	4200 / 4400	1700 770 3300	i S.p i i	i S.p i	i S.p P i C.r	p P d C.r	d i r	As the lanthanides.
91 Protactinium (231) /	Pa Pa0 <sub>2</sub>	NG CC1	1	1	1	1.	1230 /	i i	i i	d i C.r	p d C.r	(d) (r)	As the lanthanides. No stable isotopes.
92 Uranium 238 5.1	บ บכ1 <sub>4</sub> บ0 <sub>2</sub>	NG CC14	1582 / 1747	1737 / 1892	1927 /. 2060	3818 792 /	1132 590 2500	i S.p i i	i S.p d i	i S.p P i C.r	r p , p C.r	p d r	As the lanthanides.
93 Neptunium (237) /	Np Np0 <sub>2</sub>	NG CC1	/ /	1	1	3900 /	64,0 /	i i	i	i 1 C.r	r d C.r	(p) (r)	No stable isotopes. As the lanthanides.
94 Plutonium (239) 5.1	Pu PuC <sub>2</sub>	NG CC14	1207 1720	]342 1860	1507 2010	3330	641 /	i i	i i	i i C.r	r d C.r	P r	No stable isotopes. As the lanthanides.
95 Americium (241) /	Am Am0 <sub>2</sub>	NG CC14	867 /	972 . /	1100	2600	830	i .i.	i i	p i C.r	r d C.r	(p) (r)	No stable isotopes. As the lanthanides.
95 Curium (247) /	Cm CmO <sub>2</sub>	NG CC1	1	/	1	1	1340	i i	i. i	d i C.r	r d'C.r	(p) (c)	No stable isotopes. As the lanthanides.
97 Berkelium (249) /	5k Bk0 <sub>2</sub>	NG CC1	1	1	1	1	1	i i	i i	d i C.r	r d C.r	(p) (r)	No stable isotopes. As the lanthanides.

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## APPENDIX III

### OPERATING PROCEDURES FOR THE DANFYSIK 911-A ION SOURCE

The general procedure for running the source is as follows: 1) Select the oven which can give a temperature suitable to heat up the charge material in order to give a vapour pressure approximately  $10^{-2} - 10^{-1}$  torr.

- 2) Put the charge material in the selected oven and assemble the source.
- Outgas the source (filament current increasing up to approximately 20 amp DC).
- 4) Start a discharge on a noble gas, for instance argon IE.

Anode voltage on 200 V DC.

Filament 20-24 amp DC.

Apply argon (corresponding to a vacuum of  $\sim 1 - 5 \times 10^{-5}$  in the region just outside the source).

- Let the source stabilize thermally by running a discharge of approximately 2 amp anode current for a couple of minutes.
- Adjust the analyzer (the separating magnet) on the desired mass number.
- If necessary, increase the oven power (starting from zero) to obtain the needed current level.
- In most cases the argon support gas can gradually be reduced and even completely removed while simultaneously increasing the oven,

anode and filament power in order to maintain a constant discharge current.

9) Normally the most ideal way to run the source is to peak the output with all parameters.

# ION SOURCE 911-A

ELEMENT	V <sub>F</sub> fila VOLT	I <sub>F</sub> ment AMP	V <sub>A</sub> an VOLT	I <sub>A</sub> ode AMP	V <sub>o</sub> (oven) VOLT	I <sub>o</sub> (oven) AMP	I <sub>M</sub> (magnet) AMP	P <sub>o</sub> Torr	REMARKS
ARSENIC IRON SILVER COBALT BERYUIUM	4.0 5 3.2 4.8 4.3	23 28 20 24 23	45 40 70 40 45	1.3 2A 2.4 4.4 2	5.5 V 8.5 6 12 -	25 30 26 38 35	10A - 2A 10A 10A	$6 \times 10^{-6}$ 2 × 10 <sup>-5</sup> 10 <sup>-5</sup> -	∿75% of total current is single charged Main impurity iron is mass 26-27-28

# APPENDIX IV

GAS FEED SYSTEM WITH SAFETY PROCEDURES FOR HAZARDOUS AND DANGEROUS CHEMICALS<sup>(4)</sup>



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Figure AIV-1: Gas Supply System.

# See following pages for Charts of Precautions and Treatments

			HAZARD
MATERIAL		DESCRIPTION	CLASS DEGREE
Nitrogen Carbon dioxide Inert gases	N2 CO2. ~~ He, Ne, A, Kr, Xe	colorless, odorless gases	Inhalation D
Oxygen	0,	colorless, odorless gas	Flammability G
Hydrogen	Н2	colorless, odorless gas	Flammability D,M Inhalation 8
Chlorine (and HCl vapor)	C1,	pale yellow, irritating gas	Inhalation B Contact B
Fluorine (and HF vapor)	F.,	green-yellow, irritating gas	Inhalation A Contact A Flammability G
Sulfur	S ·	yellow solid	Inhalation 🛢 (fumes)
Phosphorus - Red	P	red powder or solid mass	Flammability C Inhalation C
Phosphorus - White	P	white solid	Flammability F Inhalation & Contact & Water A, B, C
Zinc	Zn	metal	None
Cadmium	Cđ	metal	Ingestion B (mild)
Gallium	Ga	metal (liquid)	None
Indium	In	metal, soft, silver	Ingestion 8
Thallium '	Tl	metal	Ingestion B (mild)
Aluminum	Al	metal	None
Carbon monoxide	co	colorless, odorless gas	Flammability D Inhalation 8
Carbon disulfide	cs,	colorless liquid, putrid odor	Inhalation Contact D
Nydrogen sulfide	H <sub>2</sub> S	colorless gas, rotten eggs odor	Flammability D Inhalation B/A
Aluminum chloride	AIC1,	yellow-white crystal	Contact B Inhalation B Water C
Silicon tetrafluoride	SIF	colorless gas	Inhalation B Contact B
Silicon tetrachloride	Sicl	colorless, fuming liquid, suffocating odor	Inhalation B Contact B Water B,C
Diborane	<sup>B</sup> 2 <sup>H</sup> 6	colorless gas, sickly sweet odor	Flammability D, I Inhalation A Contact B
Boron trifluoride	873	colorless gas	Inhalation B Contact B
Boron trichloride	BC13	colorless, fuming liquid	Inhalation B Contact B Water C
Phosphine	PH 3	colorless gas	Flammability D Inhalation A
Phosphorus pentafluoride	PF5	colorless gas	Inhalation B Contact B
Phosphorustrichloride	PC13	clear, colorless, fuming liquid	Contact B
Arsine	AsH3	colorless gas,garlic odor	Inhalation A
Arsenic trifluoride	AsF 3	oily liquid	Contact B Inhalation A Ingestion A Water B, C
Arsenic trichloride	AsC13	clear-to-pale yellow oily liquid or needle xtal	Contact B Inhalation A Ingestion A Water B, C
Stibine	<sup>SbH</sup> 3	colorless gas	Contact B Inhalation B
Antimony trifluoride	Sbr	colorless crystal mass	Contact B Inhalation B Water, C
Antimony trichloride	SbC13	colorless crystal	Contact B Inhalation B Water C
Hydrogen selenide	H <sub>2</sub> Se	colorless gas	Inhalation A
Hydrogen telluride	H <sub>2</sub> Te	colorless gas or yellow needles	Inhalation C
Lithium, sodium, potassium	Li,Na,K	silvery-white metal	Flammability 🖡 Water A
Rubidium, cesium	Rb,Cs	golden-white liquid or solid	Flammability F Water A

Γ		CLASS OF HAZARD	STATEMENTS OF HAZARD	PRECAUTIONARY	INSTRUCTIONS IN CASE OF CONTACT OR EXPOSURE
TION	A	Highly Texic	MAY BE FATAL IF SWALLOWED	Wash thoroughly after handling	R POISON & Call a Physician FIRST AIG: If swallowed, induce vomiting by sicking finger down throat or by giving mospy or strong saity water to drink. Repeat until yomit is clear. Never give anything by mouth to an un- conscious person.
INGES	в	Toxic	HARNFUL IF SWALLOWED	Wash thoroughly after handling	FIRST AID: If swallowed, induce vomiting by sticking finger down throat or by giving soapy or strong salty water to drink. Repeat until vomit is clear. Call a physician. Never give anything by mouth to an unconscious person.
TION	A	ilighly Toxlo	MAY BE PATAL IF Abcorded Through Skin	Do not get in eyes, on skin, on dothing. Wash thoroughly alter hendling.	& POISON & Call a Physician FIRST AID: In case of contact, immediately flumh syss or skin with plunty of water for at least 15 minutes while removing con- taminated clothing and shoes. Wash clothing before re-use. (Dis- card contaminated shoes.)
ASSORI	B	Toxic	HARMFUL IF AB- SORDED THROUGH SKIN	Avoid contact with eyes, skin, and clothing. Wash thoroughly after handling.	FIRST AID: In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing con- taminated clothing and shoes. Call a physician. Wash clothing before re-use. (Discard contam- inated shoes.)
	A	Highly Toxic	MAY BE FATAL IF	Do not breathe (dust, vapor, mist, gas). Keep container closed. Use only with adequate ven- tilation.	R POISON R Call a Physician FIRST AID: If inhaled, remove to fresh air. If not breathing give artificial respiration, prefer- ably mouth-to-mouth. If breathing is difficult, give oxygen.
INHALATION	8	Toxic	HARMFUL IF IN- Haled	Avoid breathing (dust, vapor, mist, gas). Keep container closed. Use only with adequate ven- tilation.	FIRST AID: If inhaled, remove to freeh air. If not breathing give artificial respiration, prefer- ably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician.
	c	Strong Sensi- tizer, Lungs	MAY CAUSE ALLERGIC RESPIRATORY RE- ACTION	Avoid breathing (dust, vapor, mist, gas). Keep container closed. Use with adequate ventilation.	Get into fresh air.
	D	Physiologic- ally Inert Vapor or Gas	(VAPOR) (GAS) RE- DUCES OXYGEN AVAILABLE FOR BREATHING	Keep container closed. Use with adequate ventilation. Do not enter storage areas unless adequately ventilated.	Get into fresh air.

	CLASS OF HAZARD		STATEMENTS OF HAZARD	PRECAUTIONARY MEASURES	INSTRUCTIONS IN CASE OF CONTACT OR EXPOSURE
	A	Corrosive, Eye	CAUSES (SEVERE) EYE BURNS	Do not get in eyes. Avoid breathing (dust, wapor, mist, gas). Xeep container closed. Use with adequate ventilation. Wash thoroughly after handling.	FIRST AID: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes, Call a physician.
CONTACT	в	Corrosive, Eye and Skin	CAUSES (SEVERE) BURNS	Do not get in eyes, on skin, on clothing. Avoid breathing (dust, vapor, mist, gas). Keep container closed. Use with adequate ventilation. Wash thoroughly after handling.	FIRST AID: In case of contact, immediately flush eyes or skin with plenty of water for at least 13 minutes while removing con- taminated clothing and shoes. Call a physician. Wash clothing before re-use. (Discard contaminated shoes.)
	с	Irritant, Eye	CAUSES EYE IRRI- TATION	Avoid contact with eyes. Wash thoroughly after handling.	FIRST AID: In case of contact, immediately flush eyes with pleniy of water for at least 15 minutes. Call a physician.
	D	Irritant, Eye and Skin	CAUSES IRRITATION	Avoid contact with eyes, skin, and clothing. Wash thoroughly after handling.	FIRST AID: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician. Flush skin with weter. (Wash clothing before re-use).
	E	Strong Sensi- tizer, Skin	MAY CAUSE ALLER- GIC SKIN REACTION	Avoid prolonged or repeated contact with skin. Wash thoroughly after handling.	FIRST AID: In case of contact, immediately wash skin with soap and plenty of water.
FLAMMABILITY	A	Extremely Flammable Liquid	EXTREMELY FLAM- MABLE	Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation.	
	B	Plammable Liquid	FLAMMABLE	Keep away from heat, sparks, and open flame, Keep container closed. Use with adequate ventilation,	
	С	Flammable Solid	PLAMMABLE	Keep away from heat, sparks, and open flame.	
	D	Flammable Gas	EXTREMELY PLAM- MABLE (GAS) (LIQUID AND GAS) UNDER PRESSURE	Keep away from heat, sparks, and open flame. Use with adequate ventilation. Never drop cylinder. Keep cylinder out of sun and away from heat.	
	E	Combustible Liquid	COMBUSTIBLE	Keep away from heat and open flame.	, e
	F	Pyroforic Substance	EXTREMELY FLAM- MABLE. CATCHES FIRE IF EXPOSED TO AIR.	Keep away from heat, sparks, and open flame. Keep container closed.	
	G	Strong Oxi- dizer	STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE	Keep from contact with clothing and other combustible materials. Do not store near combustible materials. Store in tightly closed container.	Remove and wash contaminated clothing promptly.
	H	Explosive in Air		Keep sparks or flame away.	
	1	Explosive any Conditions		Evacuate	
WATER REACTIVITY	A	Fiammable or Explosive	BURNS IN WATER OR EXPLODES	Use no water to fight fire.	
	B	Highly Toxic	FORMS POISONOUS GAS WITH WATER REACTION	See above inhalation A & B	Use no water.
	с	Toxic	FORMS BURNING GAS WITH WATER RE- REACTION	See above contact A,B,C, & D	Use no water.

## APPENDIX V

# ELECTRICAL DIAGRAMS FOR THE ION SOURCE

- 1) Ion Source Power Supply System.
- 2) Ion Source Schematics.
- 3) Telemetering System.
- 4) Safety Interlock System.
- 5) Remote Control Gas Feed System.
- 6) Remote Control Analyzer Magnet System.

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#### Power Requirements

The magnet coil mounted on the ion source housing must be connected so that the magnetic field on the axis from the filament can be cancelled out.

Filament Supply:P out  $\sim$  120-130 W (6 V/20 A and 4, 5 V/30 A)Anode Supply:250-300 V/0.1 A and 30-50 V/4-5 AOven Supply:9.5 V/30 A ( $I_{max} \sim 40$  amp.)

## Ion Source Trouble-Shooting

- If the source will not start on argon, check:
- a) Symptom: No pressure rise when the needle valve is opened.

Cause: (i) Gas bottle empty; replace with full bottle.

- (ii) Needle valve damaged and/or shaft has no connection to the needle. Repair.
- b) Symptom: Filament currents in excess of ~26-28 amp needed to start the source.
- 1. Measure the voltage over the filament feed-throughs on the ion source (switch off the extraction supply). If the resistance of the filament is <0.1-0.15 $\Omega$  at  $\sim$ 20-25 amp the filament is probably partly shorted. Replace the filament.
- 2. If there are large amounts of mass 28 and mass 14 (nitrogen), and only little or no mass 40 there probably is a leak in the gas supply system. Use a perspex or trovidur tube ∿0.5 m long (for insulating against voltage extraction) to spray the parts of the gas supply

system with gaseous helium and adjust the separating magnet to mass 4 (in this case the separator part is used as a gas leak detector!). Any leak detected must be sealed.

3. If the main contribution is mass 40, the discharge chamber is probably leaking. Check whether the springs on the gas injector are properly positioned. Check all the insulators for cracks. Check whether the gas injector, or ovens are properly placed.



Figure AV-1: Ion Source-with Power Supplies.











FROM OVEN OR FILAMENT TERMINAL 1'-1 Figure AV-3

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Figure AV-4: Filament and Oven Input Circuits

to the Telemetering System.





<u>Figure AV-5</u>: Anode Voltage and Current Input Circuits to the Telemetering System.







- A Isolation Transformer
- B Fence Door
- C Key Switch
- D High Voltage
- IS-A Isolation Area
  - V<sub>+</sub> 15 Volts DC



Figure AV-8: 150 kV Safety Interlock.

Note

Electrical diagrams of Figure 5, Remote Control Gas Feed System, and Figure 6, Remote Control Analyzer Magnet System, have been designed but have not undergone a rigorous bench testing procedure. Consequently, the schematics of the finished circuit are not included in this report.

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# APPENDIX VI

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## HEAT EXCHANGER FOR ION SOURCE



Figure AVI-1: Demineralized Water Supply.

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