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PRODUCTION OF ATOMIC OR MOLECULAR NITROGEN ION BEAMS

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#### **PRODUCTION OF ATOMIC OR MOLECULAR NITROGEN ION BEAMS\***

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#### <u>Abstract</u>

Two small ion sources have been used to generate positive nitrogen ion beams. One is a multicusp ion source, the other is a compact microwave ion source which needs no magnetic field for operation. Both sources are operated with and without a magnetic filter to control the energetic electron population near the extraction region. Results for both ion sources, including current densities and ionic species mix, are presented. The multicusp ion source can produce beams of nitrogen ions with greater than 90% of the ion species either N<sup>+</sup> or N<sub>2</sub><sup>+</sup>. The microwave ion source can generate nitrogen ion beams composed of more than 85% N<sub>2</sub><sup>+</sup> with current densities of ~ 30 mA/cm<sup>2</sup>.

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#### I. Introduction

Implantation of nitrogen ions has been used to increase the wear resistance of metals.<sup>1-4</sup> This process does not require the elevated temperatures used for thermal diffusion of nitrogen into metals; in addition, it is not a coating so it has no adhesion problems. Ion implantation can be controlled and monitored continuously, and also preferentially sputters surface protrusions, which results in a smoother surface, producing less friction for contacting surfaces such as those found on ball bearings for example.

Nitrogen ion implantation is usually carried out at energies of 10-400 kev and dose levels of  $10^{16} - 10^{18}$  ions/cm<sup>2</sup>. Ion sources that can produce nitrogen beams composed mostly of either N<sup>+</sup> or N<sub>2</sub><sup>+</sup> are preferred, because the beam does not need to be magnetically analyzed to obtain the desired ion species. On the other hand, the presence of both N<sup>+</sup> and N<sub>2</sub><sup>+</sup> in the beam results in some nitrogen atoms being deposited with either <u>half</u> or <u>twice</u> the desired energy, resulting in poor control over the implanted ion depth. Ion sources providing a high current density are also highly desirable since less time is spent per part treated for the same ion dose.

Results are presented for two small ion sources; one is a multicusp ion source<sup>5</sup>, and the other is a compact microwave ion source<sup>6</sup> which needs no magnetic field for operation. Both of these ion sources are operated with and without a magnetic filter<sup>7</sup> near the extraction plane to provide control of the energetic electron population. Ion beams with N<sup>+</sup>, or N<sub>2</sub><sup>+</sup> concentration greater than 90%, have been successfully extracted from the multicusp ion source. For the microwave ion source, N<sub>2</sub><sup>+</sup> concentrations

greater than 85% have been successfully extracted at high current densities.

#### II. Experimental Apparatus

#### A. <u>Multicusp Ion Source</u>

The multicusp ion source is fabricated from a cylindrical water cooled copper chamber (2.5 cm diameter by 5 cm long) with the open end enclosed by a two grid ion extraction system. A schematic diagram of the ion source is shown in Fig. 1. The source chamber is surrounded externally by 16 columns of small ceramic magnets to form a longitudinal line-cusp configuration for primary electron and plasma confinement. The use of 16 columns of magnets, as opposed to a smaller number, allows a larger 'field-free' region (B < 10 gauss) where the filament is placed and primary electrons are emitted. The magnet columns on the cylindrical wall are connected at the end flange by two rows of samarium cobalt magnets that are also in a line-cusp configuration.

A samarium cobalt magnetic filter near the plane of extraction divides the chamber into an arc discharge and an extraction region. The filter magnets provide a transverse magnetic field (B = 250 gauss at the center) which serves to prevent energetic primary electrons from reaching the extraction region. However, positive ions and low energy electrons can diffuse across the filter into the extraction region to form a plasma. This source can be operated both with and without the filter.

A two-electrode acceleration system is attached to the open end of the chamber. The extraction aperture is 1.6 mm in diameter giving an extraction area of  $2 \times 10^{-2}$  cm<sup>2</sup>. The source and the first or plasma

electrode are biased positive for positive ion extraction. The second electrode is electrically grounded. A plasma is produced by primary electrons emitted from a 0.5-mm-diameter hairpin tungsten filament. The chamber wall and the plasma electrode serve as the anode for the discharge. Located downstream from the second electrode is a compact magnetic deflection spectrometer<sup>8</sup> for measurement of the ion species in the extracted beam.

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#### B. <u>Microwave Ion Source</u>

The microwave ion source is fabricated from a quartz tube with a two-grid extractor at one end and the gas inlet at the other end. A schematic diagram of the source is shown in Fig. 2. The quartz tube has an outside diameter of 10 mm and an inside diameter of 8 mm. The tube is enclosed by an Evanson microwave cavity<sup>9</sup> and operated at a fixed frequency of 2.45 GHz. Microwave power as high as 500 watts can be coupled to the cavity from a Micro-Now Model 420B generator by means of a coaxial cable. Cooling air is directed at the discharge through a tube located in the body of the cavity. Additional cooling of the source is provided by an air blower. The removable end cap of the cavity allows the unit to be positioned without breaking the vacuum system. This ion source has no components of limited lifetime such as filaments or cathodes, and needs only one power supply for operation.

Ionization of the gas in the tube is initiated by a hand-held Tesla coil. When properly adjusted, the cavity will maintain a discharge in various gases at pressures ranging from a few milli-torr to several hundred torr. A tuning stub and coupling slider are provided in the cavity to properly

match the impedance of the discharge to that of the generator, and once adjusted, no further adjustments are necessary unless the flow conditions are changed. The reflected, as well as the incident microwave power, is measured using a bi-directional power meter located at the output of the microwave generator. Reflected microwave power is typically 10% of the incident microwave power when the cavity is properly tuned.

The open end of the quartz tube is enclosed by a two-electrode accelerator system. The first or plasma electrode has a 1-mm-diameter extraction aperture, and is biased at positive potential for positive ion extraction. The second electrode is connected to ground potential. Both electrodes are water-cooled. Since the quartz tube is electrically floating, the potential of the source plasma is 'tied' to that of the plasma electrode. Thus the energy acquired by the extracted ions is equal to the potential applied to the plasma electrode plus the plasma potential. As in the case of the multicusp source, a compact magnetic-deflection spectrometer is located downstream of the second electrode and is used to determine the ion species of the extracted beam and the source plasma potential.

#### III. Experimental Results

#### A. The Multicusp Ion Source

The muticusp ion source can be operated at a variety of arc voltages, arc currents, and gas flow rates. The dependence of the extracted ion species on these operating parameters has been studied in order to determine the most favorable conditions for producing either N<sup>+</sup> or N<sub>2</sub><sup>+</sup> ions. The source is operated without the magnetic filter unless specified. The arc voltage dependence of the extracted nitrogen ion species was

investigated with a constant arc current of 1.2A and a gas flow of 1 standard cubic centimeter per minute (sccm). Results, presented in Fig. 3, show that the N<sup>+</sup> concentration increases slowly with the arc voltage up to 120 V. This is to be expected since the cross-section for dissociative ionization of N<sub>2</sub>, (e + N<sub>2</sub>  $\rightarrow$  N + N<sup>+</sup> + 2e) and (e + N<sub>2</sub>  $\rightarrow$  2N<sup>+</sup> + 3e) jointly, increases faster with electron energy than simple ionization of N<sub>2</sub>, (e + N<sub>2</sub>  $\rightarrow$  N<sub>2</sub><sup>+</sup> + 2e), in the range of 30 - 120eV electron energy<sup>10</sup>.

The species dependence on arc current in the discharge was also examined for an arc voltage of 80V and a gas flow of 1 sccm. As illustrated by Fig. 4, the N<sup>+</sup> percentage increases slightly with arc current which is presumably due to the increased production of N<sup>+</sup> via two step processes such as dissociation of N<sub>2</sub><sup>+</sup>, (e + N<sub>2</sub><sup>+</sup>  $\rightarrow$  N + N<sup>+</sup> + e) or (e + N<sub>2</sub><sup>+</sup>  $\rightarrow$ 2N<sup>+</sup> + 2e), and ionization of atomic nitrogen, (e + N  $\rightarrow$  N<sup>+</sup> + 2e).

To determine the species dependence on gas flow rate, the ion source was operated with an arc voltage of 80V and an arc current of 2A. Fig. 5A is a plot of the ion species distribution in the extracted beam for various gas flow rates when the source is operated without a magnetic filter. Fig. 5B shows the case for source operation with a filter. The behavior for both cases is very similar, showing dominance of N<sub>2</sub><sup>+</sup> in the extracted beam at low pressures and N<sup>+</sup> at higher pressures. This pressure dependence can be explained in terms of the ionization mean free path of the primary electrons. As gas flow increases, and hence the gas density, the mean free path of the primary electrons becomes shorter and the discharge becomes more intense in the region of the filament. This intense discharge produces more N<sup>+</sup> via two step processes. Since the shorter mean free path of the primary electrons tends to restrict the region of maximum ion production,

keeping it farther from the extractor, the difference in ion loss rates for  $N_2^+$  and  $N^+$  may account for or contribute to the reduced percentage of  $N_2^+$  in the extracted beam:  $N_2^+$  ions are created with less energy than  $N^+$  ions<sup>11</sup>. Thus, to reach the extraction area, the  $N_2^+$  ions must survive longer than the  $N^+$  ions. However, the rate of dissociative recombination of  $N_2^+$ ,  $(N_2^+ + e \rightarrow N + N^+)^{12}$ , is much higher than the recombination rate of  $N^+$ , and may in fact be high enough to noticeably depress the amount of  $N_2^+$  ions reaching the extractor.

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The effect of the magnetic filter on the species mix, (as shown in Figs. 5A and 5B), is also dramatic. Two reasons may contribute to the large increase in N<sup>+</sup> when the filter is used. First, the filter essentially eliminates primary electrons, (and therefore direct ionization of N2), from a volume of the discharge near the extractor. This leaves a smaller source volume in which the primary electrons deposit their energy creating a more intense discharge in this region, which enhances two step production of N<sup>+</sup>. The second reason for extracting more N<sup>+</sup> is that the transport of the N<sup>+</sup> ion species across the magnetic field lines of the filter is much greater than that of  $N_2^+$ . N<sup>+</sup> ions created by dissociative ionization typically have energies of 0.25eV to several eV, while N2<sup>+</sup> is created 'cold'<sup>10,11</sup>. Thus an N<sup>+</sup> ion has a larger gyroradius and passes though the filter field more easily than an  $N_2^{+}\text{ion.}$  Hence, the reduction of  $N_2^{+}$ production combined with greater N<sup>+</sup> production and transport to the extractor, give the filter discharge a much higher percentage of N<sup>+</sup> ions for the given operating parameters.

A beam of N<sup>+</sup> ions is often more desirable than  $N_2^+$  because an ion implanter of a given energy implants nitrogen atoms from a N<sup>+</sup> ion beam

deeper into the sample than for a  $N_2^+$  ion beam. To optimize this multicusp ion source for N<sup>+</sup> ion production, a filter and a high gas flow of 4 sccm was used to enhance two step N<sup>+</sup> production; arc voltage was 124V and the arc current was 2.5A. The output signal of the mass spectrometer (Fig. 6A) shows the ion species in the extracted beam. The percentage of N<sup>+</sup> was greater than 95%. The current density was approximately 8mA/cm<sup>2</sup>. There were also some impurities in the beam associated with oxygen on the copper walls of the discharge chamber. These could presumably be reduced with the use of oxygen free copper and by baking the chamber under vacuum.

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The multicusp source was also operated to produce a high percentage of  $N_2^+$  in the extracted beam. Such a beam may be useful in situations where beam optics are particularly important. Since  $N_2^+$  is created with little transverse energy relative to N<sup>+</sup>, a  $N_2^+$  ion source would have better emittance than a similar N<sup>+</sup> ion source. In order to maximize  $N_2^+$  production and to limit N<sup>+</sup> production, the gas flow was reduced to 0.05 sccm to create a more diffuse discharge, and the arc current was reduced to 0.13A. The mass spectrum of the extracted beam is shown in Fig. 6B. The percentage of  $N_2^+$  is greater than 90% while the current density was 2.5 mA/cm<sup>2</sup>. Higher arc power levels would produce larger current densities while reducing the  $N_2^+$  concentration slightly.

#### B. <u>The Microwave Ion Source</u>

The microwave ion source can operate over a broad range of microwave power levels and gas flow rates. The dependence of the nitrogen beam species on both parameters was investigated. The microwave source was

operated at a constant gas flow of 1 sccm, and the extracted ion species distribution was studied as a function of the absorbed microwave power. Figure 7 shows that there is little variation in the beam composition with the absorbed microwave power. However, extracted current density, (presented in Fig. 8), increases linearly with the absorbed microwave power. Figure 9 shows the extracted ion species as a function of gas flow with 200 watts of microwave power. It can be seen that there is little variation in the species mix with gas flow.

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A filter has also been tested with the microwave ion source. In this case, the dependence of the nitrogen species mix on microwave power and gas flow are virtually the same as without the filter, although the extractable current density is a little lower when the filter is present.

The species mix for the microwave source is different from the multicusp source mainly because the microwave source does not have a large population of energetic electrons (~ 80 eV) as in the case of a dc filament discharge. As previously discussed, the cross section for dissociative ionization of N<sub>2</sub> increases (relative to simple ionization of N<sub>2</sub>) with higher electron energies. Thus dissociative ionization plays a smaller role in the microwave ion source, resulting in less N<sup>+</sup> production by single step processes. Also, as compared to the multicusp source, the ion production zone is fixed by the cavity position. Hence a change of gas pressure, or the addition of a magnetic filter field, has less effect on positive ion extraction in the microwave ion source.

The microwave ion source produces a high percentage of  $N_2^+$  for most operating conditions. To maximize the  $N_2^+$  percentage the source was operated with 1 sccm of gas flow and 300 watts of power without the

magnetic filter. Fig. 10 shows the output of the mass spectrometer which details the extracted ion species. Here, the percentage of  $N_2^+$  was greater than 85% with an extracted current density of 29.8 mA/cm<sup>2</sup>. The impurity content in the beam was almost negligible.

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#### IV. Conclusion

Results for operation of two types of ion sources with nitrogen gas have been presented. The multicusp ion source can produce beams of nitrogen ions with greater than 95% N<sup>+</sup> at a current density of 8mA/cm<sup>2</sup>, or greater than 90% N<sub>2</sub><sup>+</sup> at a current density of 2.5 mA/cm<sup>2</sup>. In order to generate a high N<sup>+</sup> concentration, the use of a magnetic filter was found to be advantageous with this source. The microwave ion source, on the other hand, can produce nitrogen ion beams with greater than 85% N<sub>2</sub><sup>+</sup> at a current density of ~ 30 mA/cm<sup>2</sup>. No magnetic multicusp fields were used in this case.

Both of these sources have unique properties that make them well suited as ion sources for nitrogen ion implantation. The multicusp source produces very high percentages of either N<sup>+</sup> or N<sub>2</sub><sup>+</sup> at modest current densities. This ion source can be easily scaled up to produce large diameter beams and high ion currents. The microwave source produces a high percentage of N<sub>2</sub><sup>+</sup> at high current densities. Operation of this source is extremely simple and reliable with very low beam energy spread<sup>13</sup>. Magnetic separation of the beam from either ion source can be avoided since the extracted beam is made up almost entirely of a single ion species.

### **Acknowledgments**

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#### **Figure Captions**

- Fig. 1 Schematic diagram of the multicusp ion source.
- Fig. 2 Schematic diagram of the microwave ion source.

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- Fig. 3 A graph of the extracted nitrogen ion beam composition as a function of the arc voltage in the multicusp ion source. Gas flow was 1 sccm and arc current was 1.2 A.
- Fig. 4 A graph of the extracted nitrogen ion beam composition as a function of the arc current in the multicusp ion source. Gas flow was 1 sccm and arc voltage was 80 V.
- Fig. 5 A graph of the extracted nitrogen ion beam composition as a function of the gas flow in the multicusp ion source. Arc voltage was 80V and the arc current was 2A. Fig 5A shows the case for operation without a magnetic filter and Fig. 5B the case with the filter.
- Fig. 6 Spectrometer output signal showing the beam species from the filtered multicusp ion source. Fig. 6A is the case for operation with an arc voltage of 124V and an arc current of 2.5A with a gas flow of 4 sccm, and shows N<sup>+</sup> composing ~ 97% of the nitrogen species. Fig. 6B is the case for operation with an arc voltage of 124V and an arc current of 0.13A with a gas flow of 0.05 sccm, and shows N<sub>2</sub><sup>+</sup> composing ~ 93% of the nitrogen species.
- Fig. 7 A graph of the extracted nitrogen ion beam composition as a function of the absorbed microwave power. Gas flow was 1 sccm.
- Fig. 8 A graph of the extracted nitrogen current density as a function of the absorbed microwave power. Gas flow was 1 sccm.

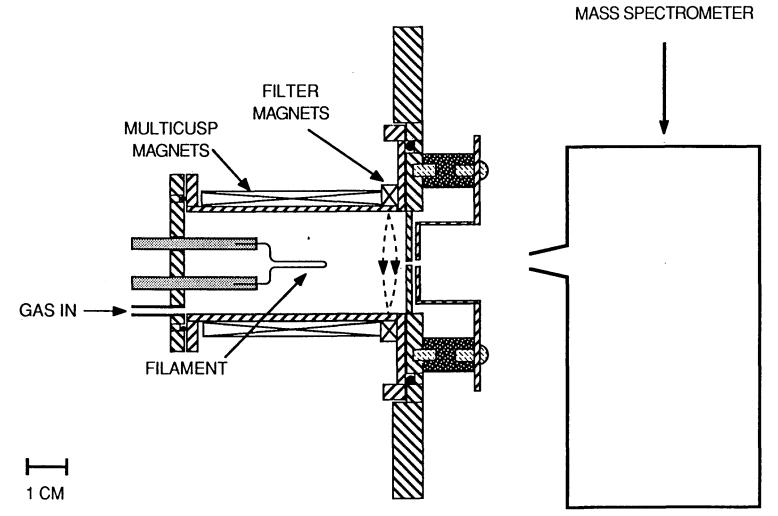
- Fig. 9 A graph of the extracted nitrogen ion beam composition as a function of the gas flow in the microwave ion source with 200 watts of microwave power.
- Fig. 10 Spectrometer output signal showing the beam species from the microwave ion source.  $N_2^+$  composes ~ 87.5% of the nitrogen species. Microwave power was 300 watts with a gas flow of 1 sccm.

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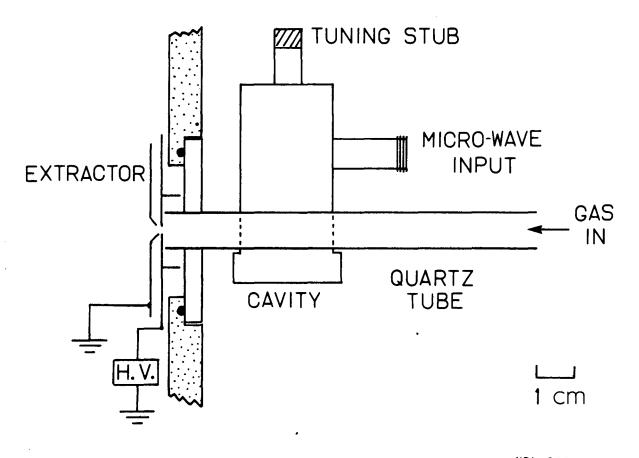
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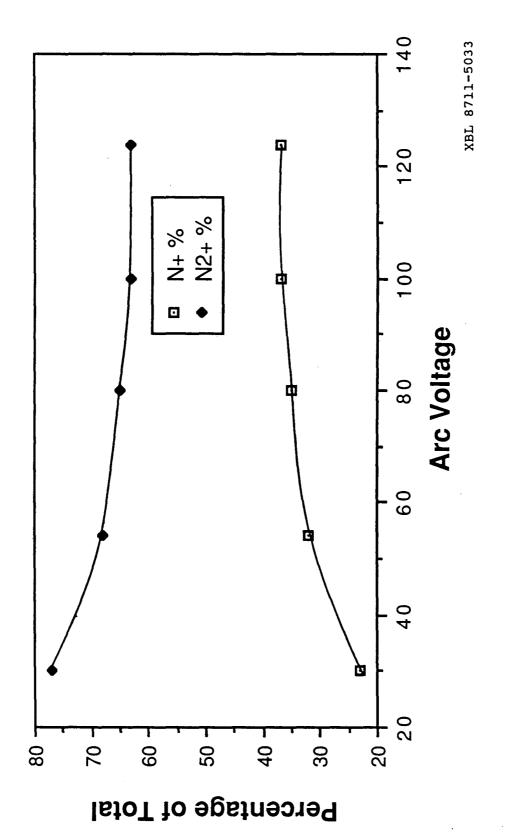


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Fig. 2

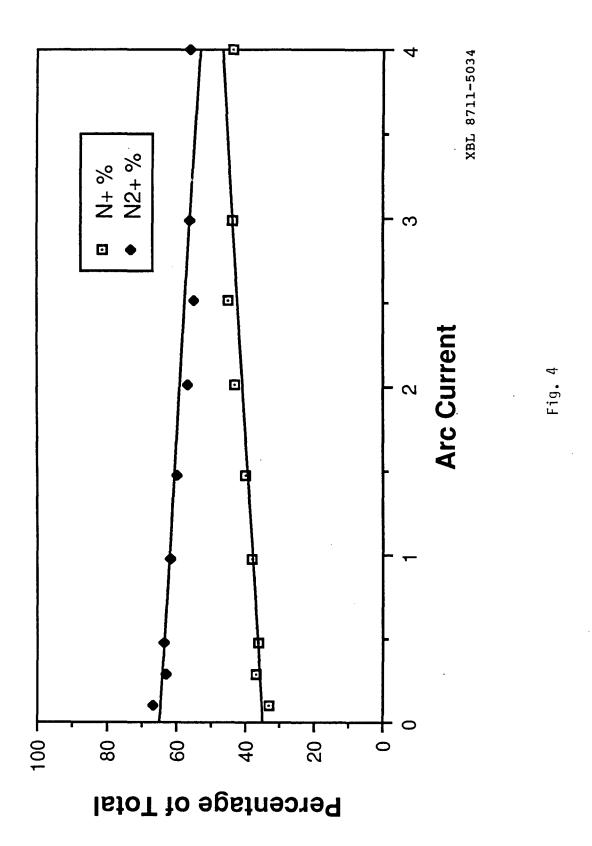


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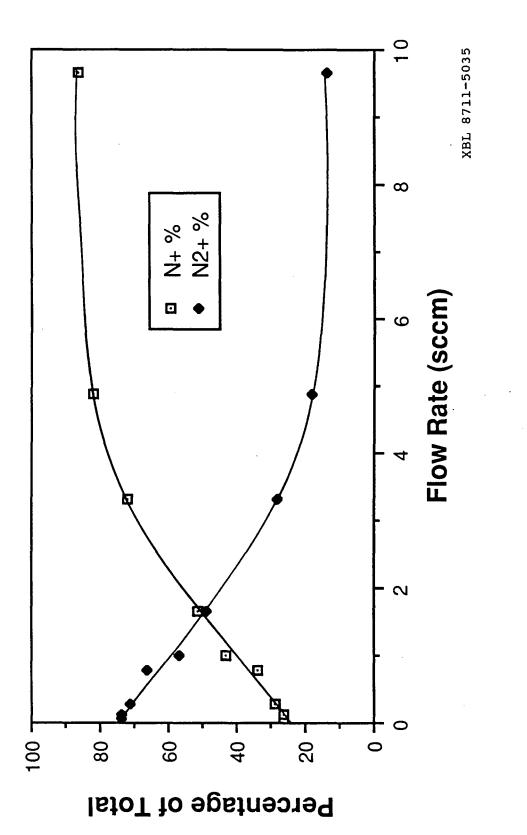
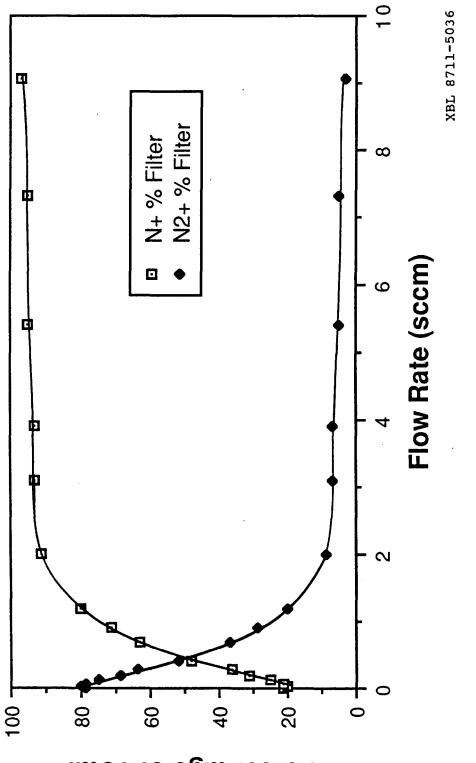


Fig. 5a



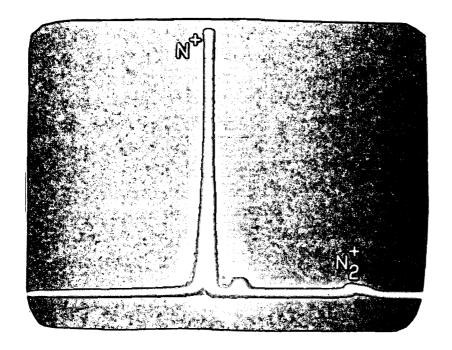
Percentage of Total

Fig. 5b

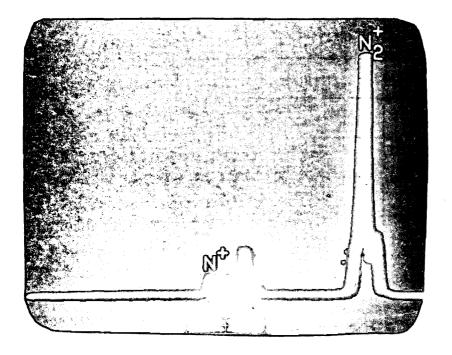
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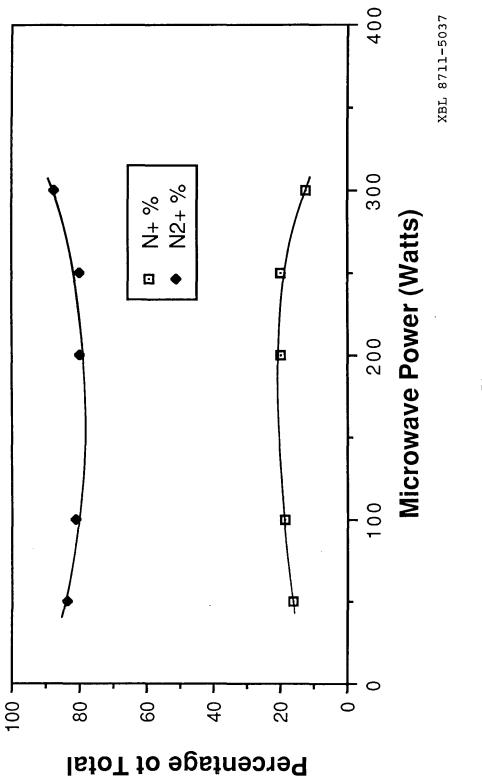


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Fig. 6



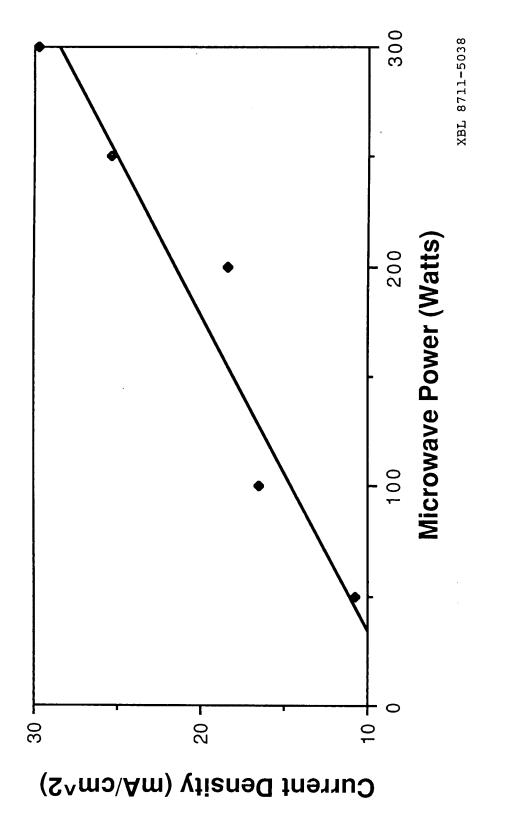


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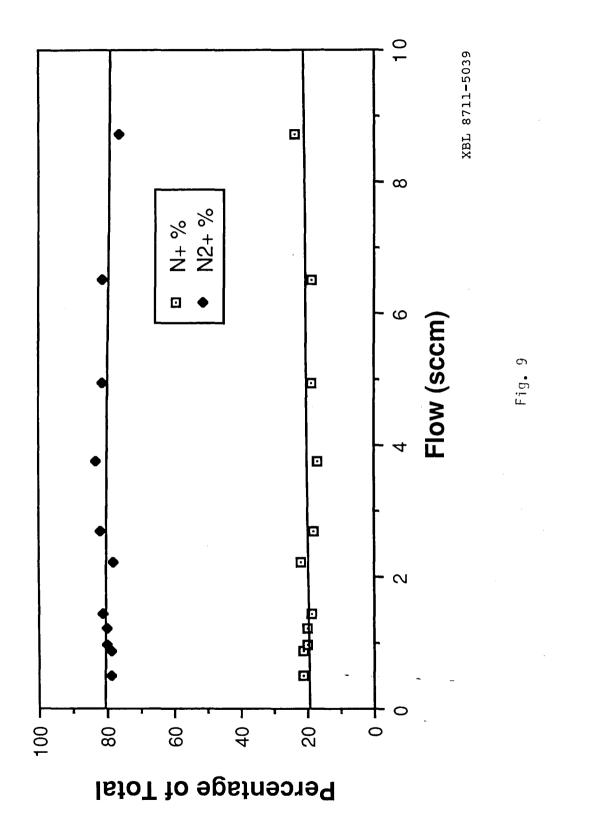
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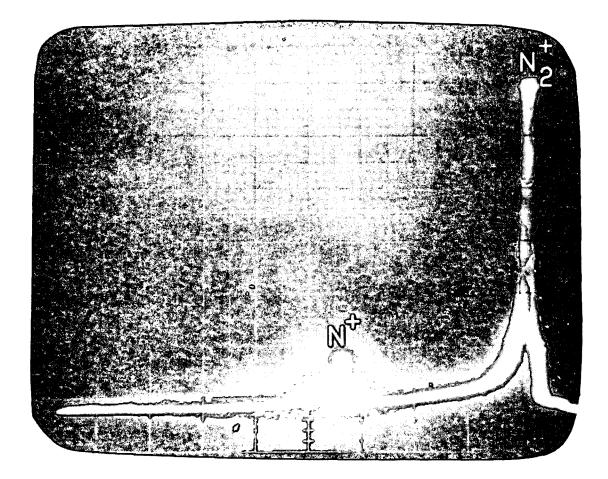




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Fig. 10

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