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Interim Report
July 1980

DIRECT-WRITE ION LITHOGRAPHY

Hughes Research Laboratories

R.P. Vahrenkamp

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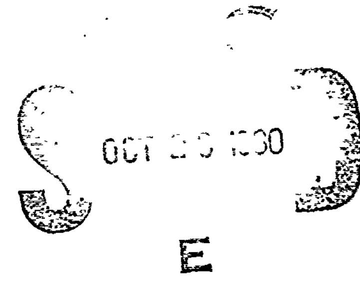

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For a practical ion-lithography column of 150 kV, the light elements Li, Be, B, C, and Si were found to be the most promising for resist exposure. With the exception of Li, these elements emit doubly charged ions when used on a liquid metal (LM) ion source. Lithium, although highly reactive, can be operated as a single-element species, while the others must be used in alloys. Mass spectra for alloys of

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Be-Au, Si-Au, and B-Pt were obtained and showed significant fractions of the desired ion species.

A new LM ion source structure (needle and heater assembly) was tested and found to improve the stability of the Be-Au ion emission. This ion source structure appears to be suitable for all elements and alloys considered for resist exposure.

PMMA exposures were made using flood and focused ion beams. Si^+ and Be^+ range measurements in PMMA were made after exposure in a conventional ion-implantation system. The results show that 0.5 µm of resist can be exposed with Be^+ ions at an energy of 60 to 80 keV while the same exposed depth requires 175 to 200 keV for Si^+ ions. Focused-beam exposures in PMMA were made using the Be_{40}-Au_{60} alloy as the ion source material. Since the focused beam was not mass separated, the gold was implanted along with the beryllium. Because of the short range of the gold ions, polymerization of the resist occurred at doses greater than $\sim 9 \times 10^{12}$ Be^{++}/cm^2. At lower doses, the exposed linewidths were 3000 to 4000 Å, in good agreement with gallium operation. Preliminary measurements indicate an exposed depth of ∿3000 Å for a development time of 60 sec. Thus, the low Be^{++} dose at 55 kV is not sufficient for maximum range, and most of the exposed depth is due to the combination of Be^+ and Be^{++} ions.

TABLE OF CONTENTS

SECTION		PAGE
1	INTRODUCTION	4
2	SUMMARY	7
3	TECHNICAL PROGRESS ON TASK 1	9
	A. Optimum Ion Species	9
	B. Ion Source Fabrication	19
	C. Resist Study	23
4	CONCLUSIONS	33
	REFERENCES	34

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SECTION 1

INTRODUCTION

This is the first interim technical report in the Direct-Write Ion Lithography program. The overall objective of this program is to conduct a theoretical and experimental study directed toward utilizing liquid metal ion sources in an ion-beam accelerating and focusing column that is designed for performing direct-write ion-beam lithography. The program is directed into four technical tasks:

(1) Optimal ion source for resist exposure

(2) Electrostatic scanning

(3) Alignment techniques

(4) Ion lithography column design.

Task 1 will determine the optimal ion source for direct-write ion lithography. The optimal ion species and energy for resist exposure will be investigated. Resist-exposure experiments will be performed with candidate liquid-metal ion sources and focused ion beams, as well as with ion-transmission masks and conventional ion sources. In parallel with the resist-exposure investigation, the technology of liquid-metal ion sources will be explored to determine which ion species can be produced. From these, the ion species that is optimal for resist exposure will be chosen. A liquid-metal source of the chosen ion species will be fabricated to demonstrate focused-ion-beam resist exposure. Sample exposures will be delivered to RADC/ESE.

Task 2 will investigate the maximum "distortion-free" electrostatic scan field. A theoretical analysis will be made to determine the dynamic correction signals that can be applied to the final-focusing lens and the deflector to minimize the deflection aberrations. The size of the scan field in our focusing column will be measured with and without application of the most significant (field curvature) dynamic correction. Task 3 will investigate alignment techniques for direct-write ion lithography. Experiments will be performed to evaluate the use of

secondary-electron detection and of photon detection for the location of alignment marks by the focused beam. Task 4 will develop the design of an ion-focusing column and interface that can replace the E-beam column on the existing RADC/ESE E-beam mask fabrication system. A theoretical ion-optical design will be made of the column elements. Then mechanical designs of the column and interface will be developed and layout drawings prepared. The column and interface electronics will be specified in block-diagram form. A design review will be held, and, after approval has been obtained, the cost of constructing the focusing column and interface will be estimated.

The tasks will be pursued over a 30-month period in accordance with the proposed phasing schedule, shown in Figure 1. Consequently, the first 15 months of the program deal solely with Task 1. At the end of this period, the remaining tasks will begin in parallel.

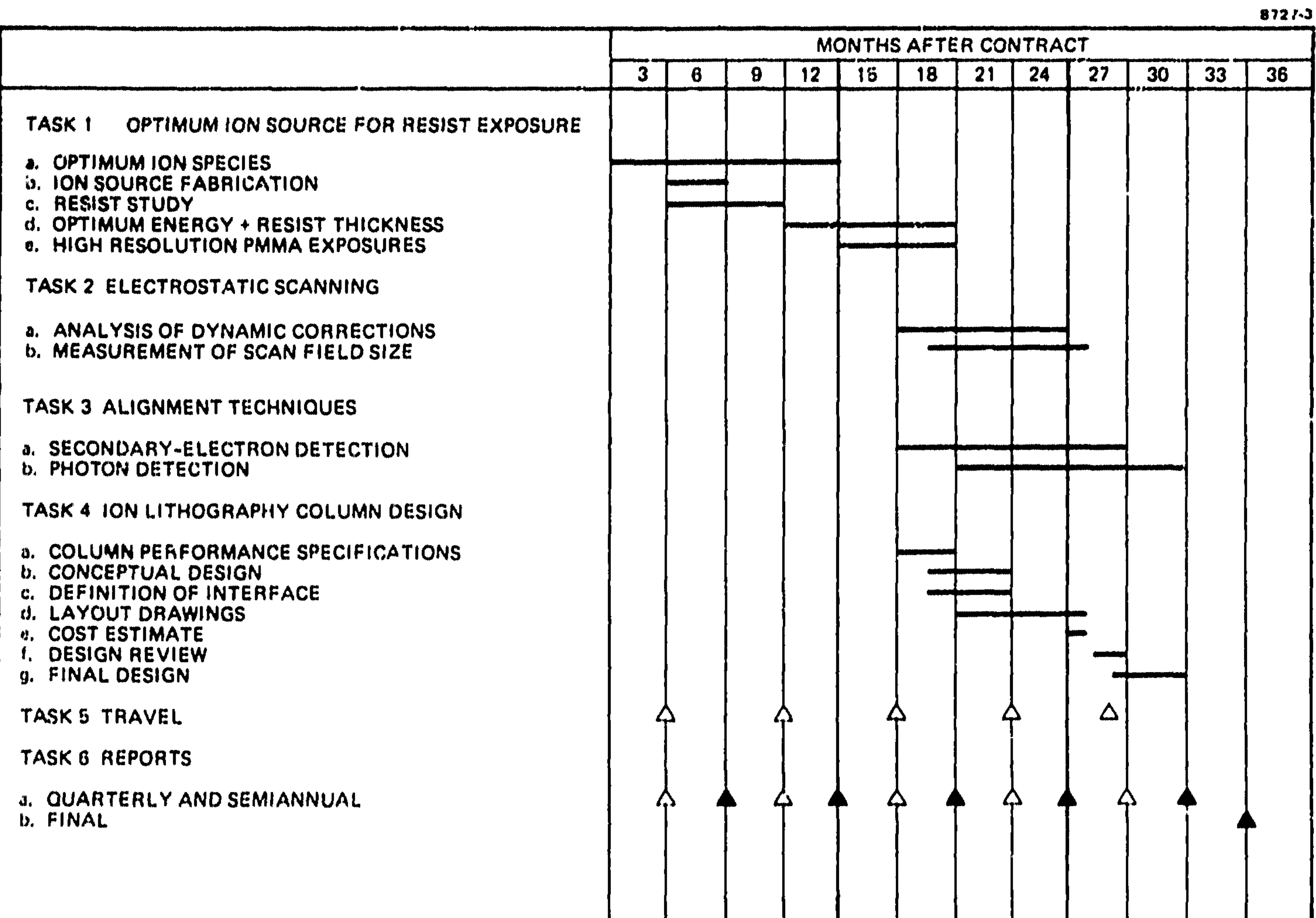

Figure 1. Phasing schedule.

SECTION 2

SUMMARY

The past two quarters of the Direct-Write Ion Lithography program were devoted to an investigation of optimum ion species, ion sources, and ion resist exposures. Since a practical ion focusing column will be limited to ~150 kV, only singly charged ions up to mass 20 (neon) and doubly charged ions up to mass 31 (phosphorus) were considered adequate to expose resist thicknesses of 0.5 µm or more.

In addition to range considerations, candidate ion species must be extracted from materials (in elemental or alloy forms) that have low vapor pressure and are liquid metals. Of the single elemental materials, only lithium appears compatible with liquid-metal ion-source operation (i.e., low vapor pressure at its melting point). The main disadvantage of lithium is its extreme reactivity in a non-inert atmosphere. Because of this great propensity for contamination, lithium may prove to be unsuitable as a practical material for an ion focusing column.

Several alloyed materials for ion-source operation were also tested. The use of alloys enlarges the number of elements that can be ionized by producing suitable molten metals from elements that individually have prohibitively high melting points and/or vapor pressures. These include Au-Be and B-Pt, in addition to the previously investigated Au-Si. Au-Be is a good choice since a high percentage of beryllium can be used (~50 at.%) and because the emitted beryllium ions are mainly doubly charged (for maximum range). The B-Pt eutectic was also investigated as a possible source material for resist exposure. Although the range of boron in resist is good (~1 µm at 150 kV), the use of a dopant ion is probably not optimum if the range of the ions transmitted into the substrate is significant. The effects on the substrate of impurity deposition and crystal damage are problem areas that will be investigated for all ion species under consideration. For silicon substrates, contamination can be eliminated by using Si^{++} ions. The Au-Si eutectic alloy, used previously at HRL to produce Si^{++}, is considered a good candidate for resist exposure. Another possible alloy for resist

exposure is the C-La eutectic. Carbon ions are mostly emitted doubly charged and would thus have sufficient range. Again, the effect of carbon impurities, if any, will need to be investigated.

Ion exposure of PMMA was begun using both flood and focused ion beams. Since Be and Si ions appear to be two promising candidates for resist exposure, the range of these two ion species in PMMA was determined.

Using the conventional HRL 300-kV ion-implantation machine, PMMA exposures were made through a 1.0-mm-diameter pinhole for both Be^+ and Si^+. For Si^+, the beam energy was varied from 150 to 250 keV while for Be^+ the beam energy was varied from 50 to 100 keV. For both species, doses in the range of 1 to 3×10^{13} ions/cm^2 were used. The results show that, for singly charged Be ions, a 0.5-μm depth of resist can be exposed with a beam energy of 60 to 80 keV, depending on dose, while the same thickness of resist can be exposed with Si^+ ions in the range of 175 to 200 keV, again depending on dose.

PMMA exposures were also made in the single-lens focusing column using the Au-Be ion source. The resolution of the focused spot appeared to be comparable to that of gallium. Lines 100 μm long were exposed with the focused beam at doses ranging from 3 to 30×10^{12} Be^{++}/cm^2. At doses above $\sim 9 \times 10^{12}$ Be^{++}/cm^2, the gold ions began polymerizing the resist, which caused the lines to remain undeveloped. Linewidths varied from 3000 to 5000 Å depending on dose and beam current. An investigation of depth profiles is presently underway.

SECTION 3

TECHNICAL PROGRESS ON TASK 1

A. OPTIMUM ION SPECIES

Work during the past two quarters was devoted to an evaluation of various ion species that could be used in an ion-lithography column. Although protons would be a good choice for such a system, no liquid-metal source of protons is presently available. The liquid metal ion source, on the other hand, is capable of generating a wide variety of other species. It is a relatively simple source, can be repeatedly vented to atmosphere with no deterioration, and does not require an ultra-high-vacuum system. The liquid-metal source has demonstrated very small focused spots with high current density (400-Å diameter at 5 A/cm^2 has been achieved at HRL). A disadvantage of this type of source is that it does not operate on gaseous or high-vapor-pressure materials.

A practical ion-lithography column should have an operating voltage that does not exceed 150 kV. Beyond this range, high-voltage standoff, arcing damage, and component isolation become more of a problem. This voltage constraint implies that only light elements can be used to penetrate and expose any reasonable thickness of resist. Resist-pattern features with depth-to-width aspect ratios greater than 2:1 are desired. Since spot diameters in the range of 1000 to 2000 Å are anticipated, the resist thickness should be ~5000 Å. Theoretical ranges are given in Ref. 1 for various ions in C_2OH_4 (representative of PMMA). For singly charged ions, elements with mass 20 (Ne) or less should be used, while doubly charged ion species can extend to approximately mass 31 (P). Primary considerations in choosing an ion species for molten sources are the vapor pressure and melting point of the material. If the elemental vapor pressure is high, alloying the material with other metals is often appropriate and can result in significantly lower operating temperatures. The remainder of this section discusses the various ionic species appropriate for an ion-lithography column, both single element and multielement (alloyed), and lists the advantages and disadvantages of each.

1. Single-Element Materials

Of the light elements ($Z \leq 31$), only lithium appears to be feasible as a single-element species. Its melting point and vapor pressure are both low (180°C and 1×10^{-10} Torr, respectively), and it has been used by other workers.[2] They generated a beam consisting mainly of singly charged lithium with small amounts of Li_2^+ and Li_3^+. Figure 2 (taken from Ref. 2) shows a typical lithium spectrum. Lithium's extremely high reactivity — its main disadvantage — requires that it be kept in a clean, inert atmosphere at all times. This has serious implications for column design since additional column pumping and vacuum isolation would be required. An appropriate facility for preparing the source and transferring it to the column would also be necessary. Although alloying lithium with other materials (e.g., gold) might reduce its reactivity, other alloyed materials appear to be more attractive. Other light single elements — Be, B, C, Mg, Si, P — all have high vapor pressures at their melting points, while the remainder (up to mass 31) are emitted as singly charged ions and thus would not have sufficient range at 150 kV.

2. Alloyed Materials

Alloyed materials extend the number of ion species that can be used in an ion-lithography system. The undesired ions can then be mass separated in the column and only the light ions transmitted for resist exposure. The use of heavy alloying elements (e.g., gold or platinum) would simplify mass separation and thus reduce the size of the mass separator needed. It is also possible to utilize the total unseparated beam to expose resist. First, the range difference between the light and heavy ions can be as much as an order of magnitude, and over-exposure by the heavy ions will be limited to the first few hundred angstroms of resist. Second, since the secondary electrons produced by the heavy ions should be short ranged (less than 50 Å), line broadening in this top layer should not be appreciable. Third, over-exposure of PMMA by a factor of 2 to 4 by the heavy ion should not be sufficient to polymerize the resist.

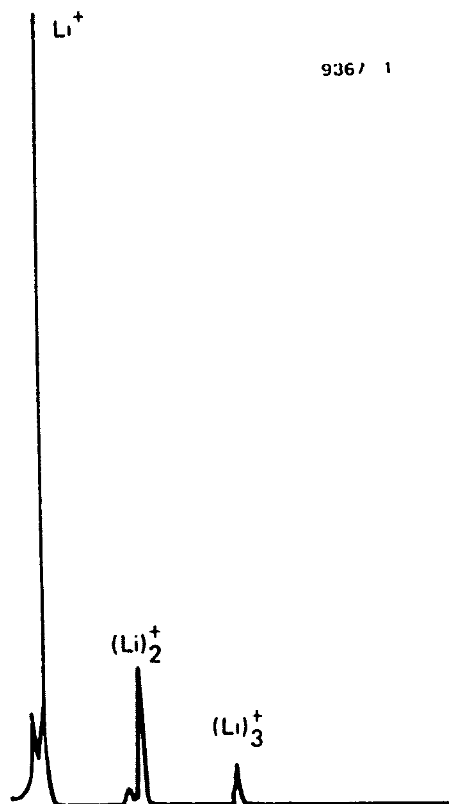

Figure 2. Mass spectrum of ions from liquid lithium.

The evaluation of alloy materials involves preparing the alloy, wetting the needle structure with the alloy, and then operating the ion source in the einzel-lens column to obtain a mass spectrum. A schematic of the einzel lens focusing column with the ExB separator is shown in Figure 3.

a. Beryllium-Gold

A partial phase diagram from Hansen[3] for the Au-Be system is shown in Figure 4. Since the vapor pressure of beryllium is not appreciable at 800°C, beryllium concentrations of up to 50 at.% appear feasible. Two compositions of the alloy were made; the first contained 40 at.% Be and had a melting point of ∿600°C, while the second consisted of 50 at.% Be and had a melting point of ∿750°C. Mass spectra for both alloys were obtained using the einzel lens focusing column; the results are shown in Figures 5 and 6. The Be^{++} ion current comprised ∿30% of the total current for the first alloy and ∿42% for the second alloy. Thus, the Be-Au alloys with their moderate operating temperatures and high Be^{++} percentages appear to be particularly attractive for resist exposure.

Although the alloys appeared to wet the needle structure properly, the source operation was erratic when compared with the standard gallium ion source. Thus, an investigation into alternate source structures was begun; the new source is discussed below.

b. Gold-Silicon

The use of the gold-silicon eutectic alloy as a source material was investigated during the past year. We found it to operate satisfactorily with spot sizes comparable to those of gallium. The mass spectrum, given in Figure 7, shows that an appreciable portion of the total beam current is Si^{++}. The phase diagram for the Au-Si system, given in Figure 8, shows that the silicon content can be increased to ∿45% at 1000°C. No problems are expected in operating at this temperature, and tests are planned to investigate this higher-Si-ratio alloy. The main advantage in using silicon is that it will not introduce additional impurities into silicon substrates. In addition, since the

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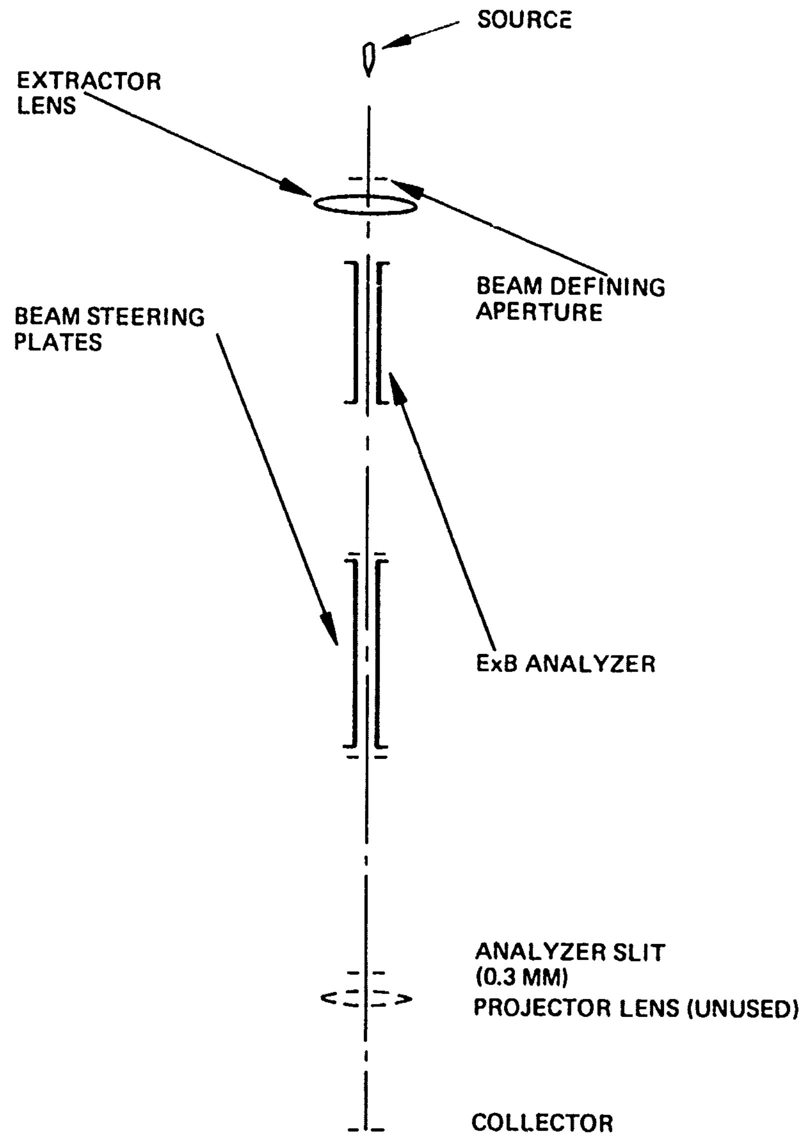

Figure 3. Mass separation experiment using an ExB mass separator in the double-einzel lens column.

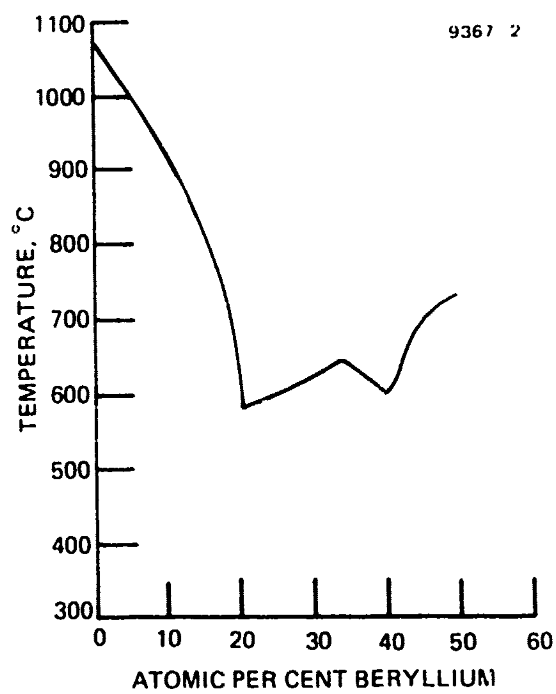

Figure 4. Au-Be phase diagram.

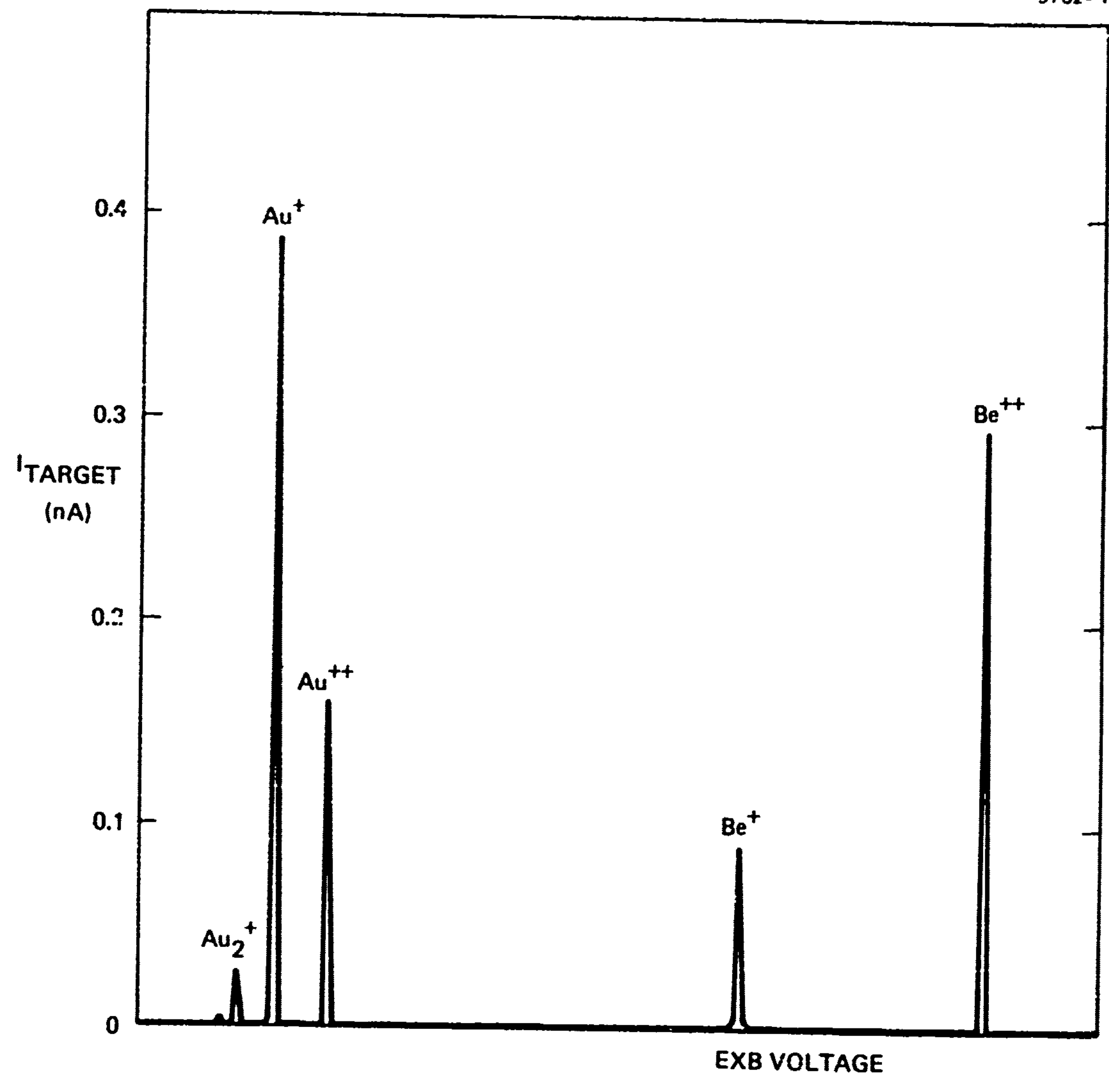

Figure 5. Mass spectrum of liquid-metal ion source using Au_{60}-Be_{40} alloy.

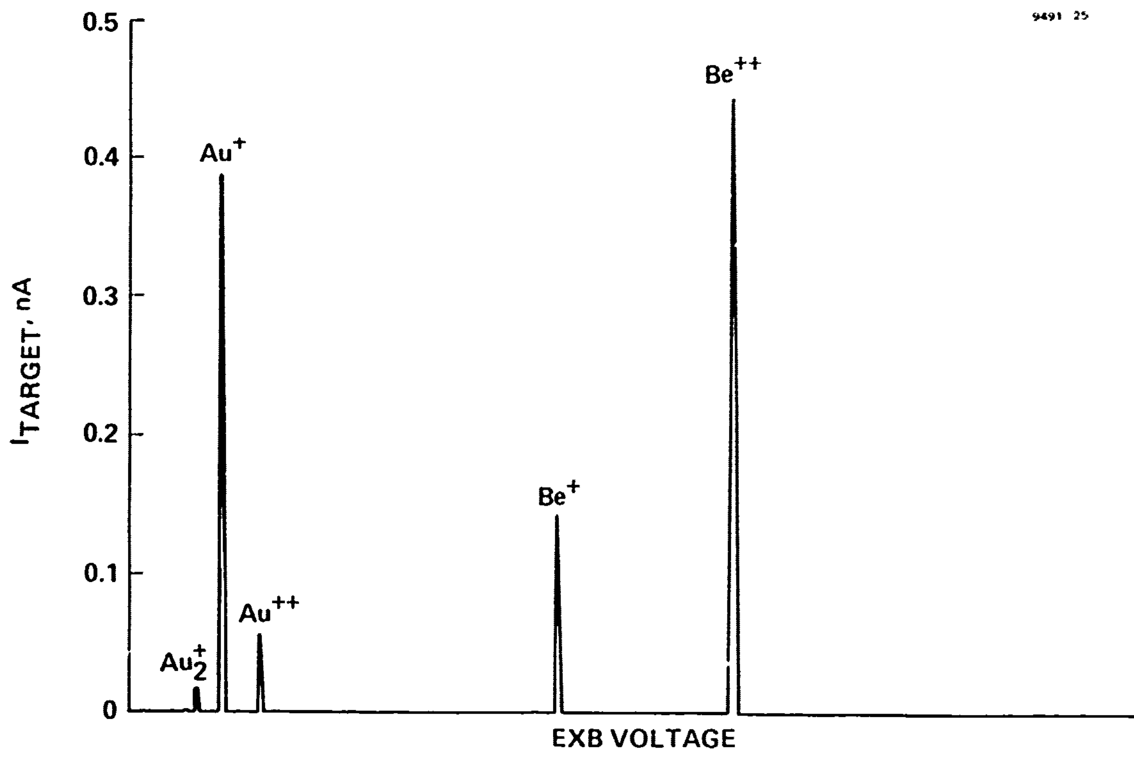

Figure 6. Mass spectrum of liquid-metal ion source using Au_{50}-Be_{50} alloy.

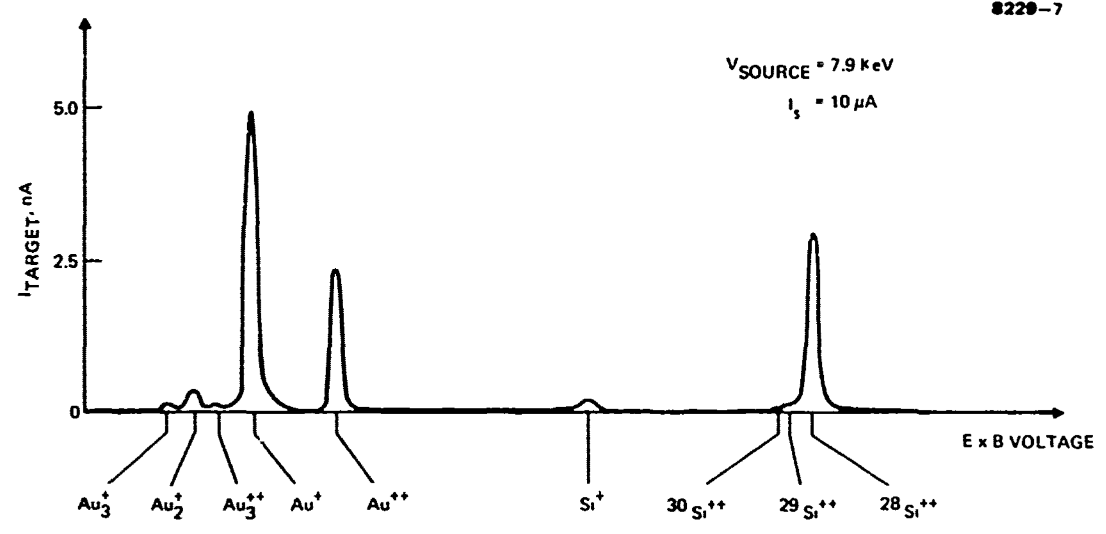

Figure 7. Mass spectrum of Au-Si beam from a liquid-metal source.

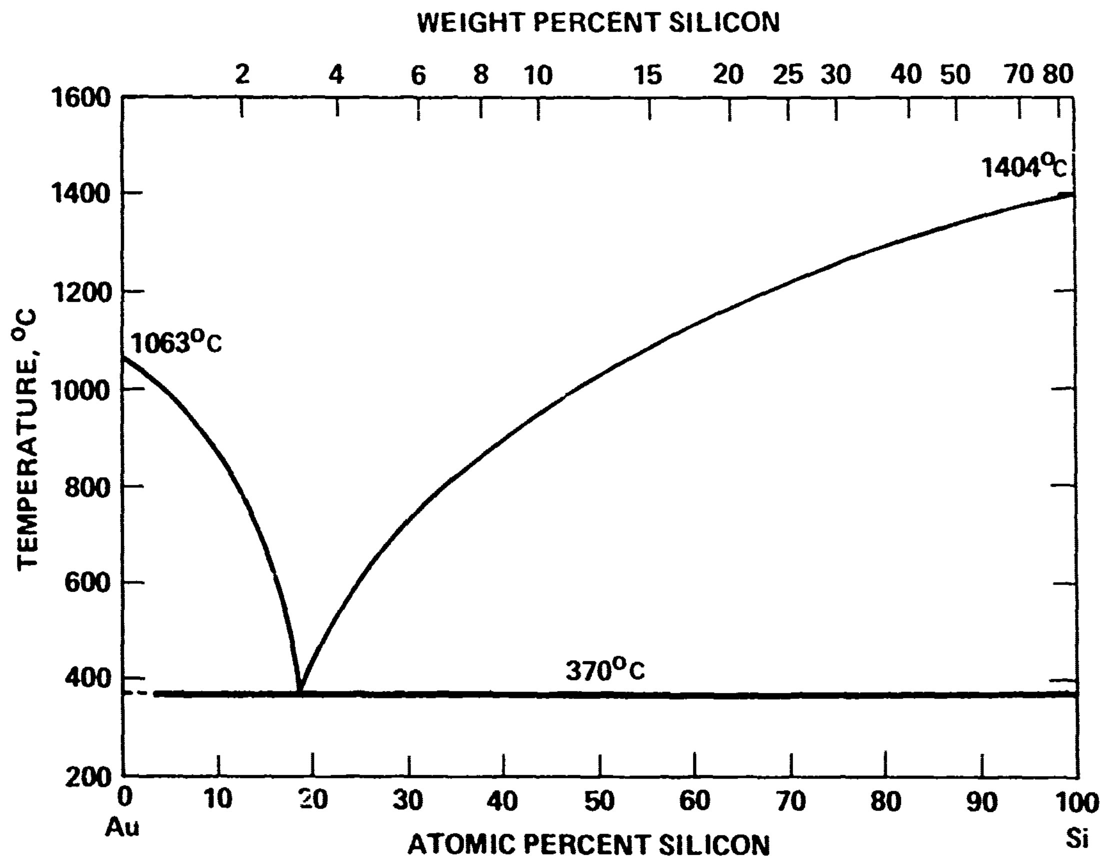

Figure 8. Phase diagram for Si-Au.

majority of the silicon ions are doubly charged, a 150-kV column should be adequate for a resist thickness of 0.5 μm.

c. Boron-Platinum

We recently investigated the B-Pt eutectic alloy as a source material for boron doping and as a possible candidate for resist exposure. The phase diagram[4] for B-Pt is shown in Figure 9, and a mass spectrum of $B_{28}Pt_{72}$ is shown in Figure 10. An interesting aspect of this system is that it can be used for simultaneous resist exposure and doping of the underlying substrate. In most practical applications, however, since any introduction of dopant ions into the substrate would not be favorable, the use of boron would be limited.

d. Carbon-Lanthanum

An interesting alloy for producing carbon ions is the La-C system; a phase diagram[3] is shown in Figure 11. Approximately 15 at.% carbon appears feasible with the carbon being emitted primarily as doubly charged ions and the lanthanum mostly as doubly and triply charged ions.[5] An alloy will be prepared during the next quarter and tested on a standard ion source structure. A mass spectrum will also be obtained.

e. Gold-Lithium

Alloying lithium with gold may be a technique for reducing the reactivity of lithium. Approximately 15 at.% gold can be added to lithium before the temperature for lithium evaporation becomes significant (∿300°C). If handling pure lithium proves to be too cumbersome, this alloy will be investigated in more detail.

B. ION SOURCE FABRICATION

Since a new ion source will be fabricated under Task 1, we began an investigation of a needle and heater geometry optimized for the alloys and elements used for resist exposure. In general terms, the critical voltage at which ion emission first occurs from a LM source is dependent on electrode geometry and surface tension of the

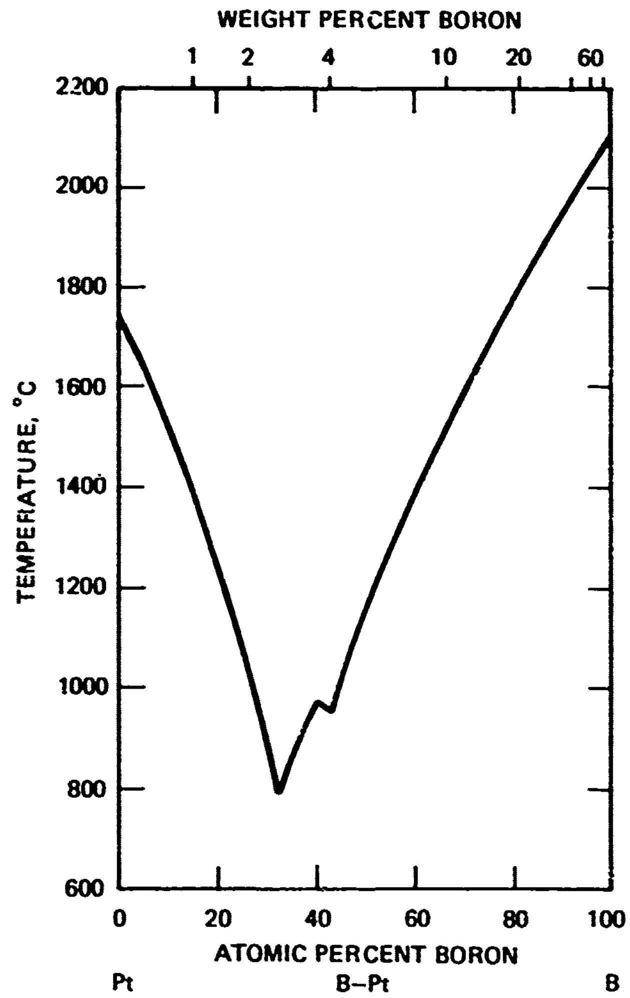

Figure 9. Phase diagram for boron-platinum.

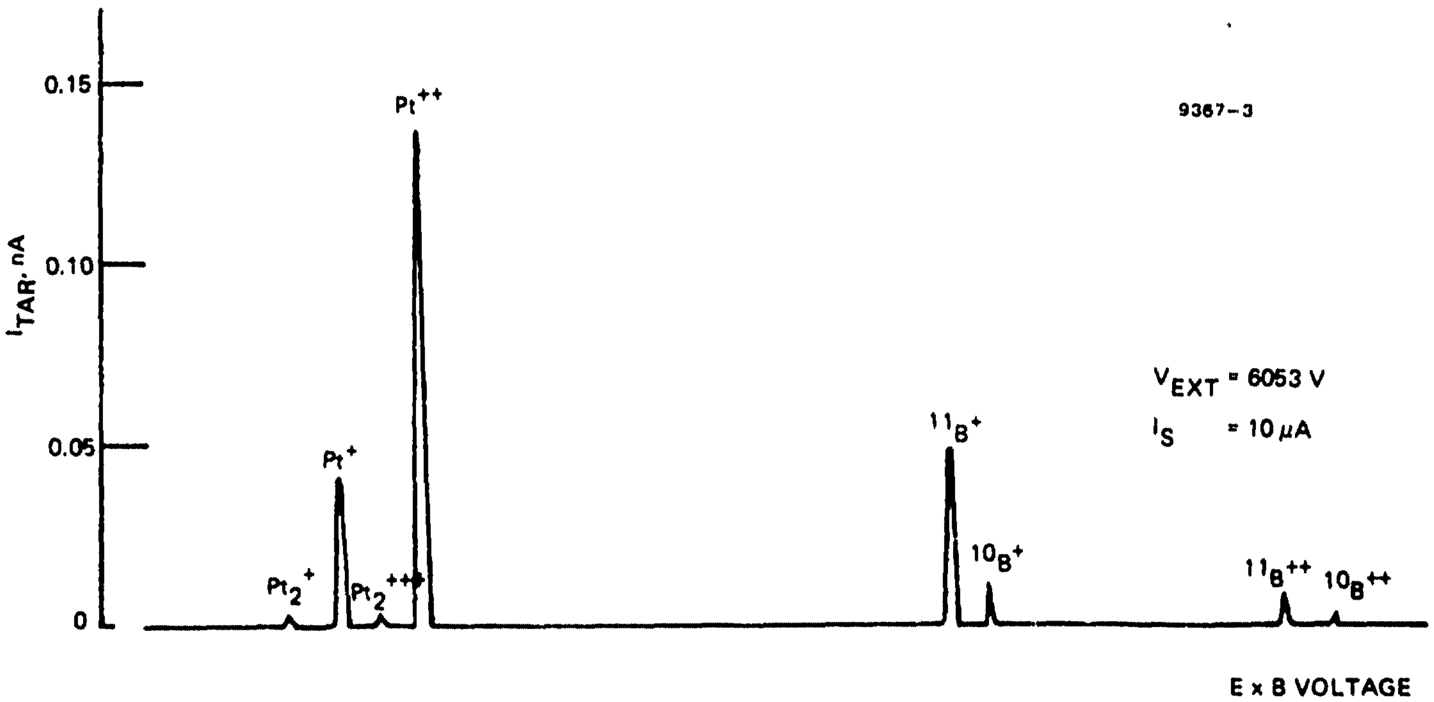

Figure 10. Mass spectrum of B-PT beam.

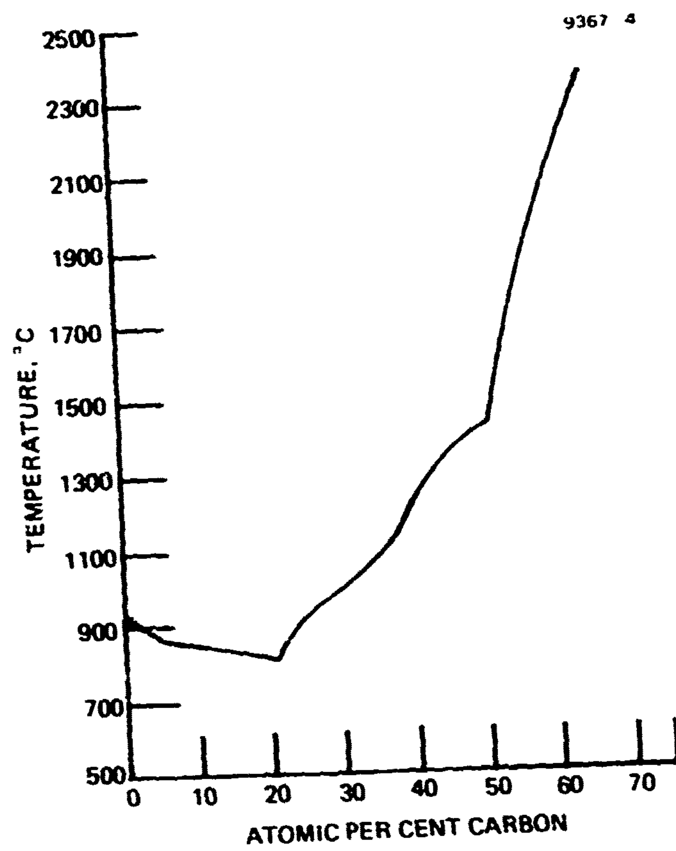

Figure 11. C-La phase diagram.

liquid material. The formation of a Taylor cone, field desorption, and field ionization have been discussed by others[6] and are felt to be the basic processes involved in LM ionization. However, several other factors must be considered for long-term stability of an ion source when in use in a practical focusing column; these include:

- Ambient vacuum conditions, including backsputtering effects
- Needle size
- Reservoir geometry and position with respect to the needle
- Reactivity of the liquid metal with the needle, particularly at higher operating temperatures.

As mentioned above, the Au-Be alloy is not as stable as Ga when operated on the standard HRL ion-source structure. At low currents (below $\sim$20 μA), the source extinguishes after several minutes of operation and then requires a higher extraction voltage to re-start. This type of operation suggests an interruption in the flow along the needle shaft and appears to be common to the high-surface-tension materials. The new needle-heater geometry shown in Figure 12 was fabricated. This geometry provides an adequate reservoir of molten metal near the needle's tip and is also very efficient in terms of power requirements. The best results with this geometry were obtained when the diameter of the needle was reduced from 0.38 mm to 0.15 mm. Although the new structure has not been operated in the focusing column, the tests to date show a marked improvement in source stability. Tests will continue during the next quarter to better characterize the source and to determine its compatibility with other materials that will be used for resist exposure.

C. RESIST STUDY

Ion exposure of PMMA was begun during the past quarter using both flood and focused ion beams. Since silicon and beryllium ions appear to be two promising candidates for resist exposure, the tests were conducted using Si^+ and Be^+ for the flood beam experiments and the Au-Be alloy for the focused beam experiments.

Figure 12. Needle and heater arrangement for new ion source structure.

Using conventional HRL ion-implatnation equipment, PMMA exposures were made through a 1.0-mm-diameter aperture for both Si^+ and Be^+. In the case of Si^+, the beam energy varied from 150 to 250 keV with Si^+ and from 50 to 100 keV with Be^+. The doses for both cases were in the 1 to 3 x 10^{13} ions/cm^2 range. The results are shown in Figures 13 and 14, where the depth of exposed resist is plotted first as a function of beam energy and then as a function of dose. For reference, some previous HRL proton exposures are also shown. The developer was 30% isopropyl alcohol: methylisobutylketone for all exposures, and the development time was 60 sec for all except one. The exception was the 1 x 10^{13} H^+/cm^2 exposure, which had a development time of 360 sec. The depth measurements were made by running the stylus of a Sloan Dektak across each exposed spot. For the Be^+ and Si^+ exposures, development times greater than 60 sec did not appear to result in any significant increase in exposed depth. Thus, by proper selection of energy and dose, the depth of exposed resist can be controlled over a fairly wide range.

Using the Be_{40}-Au_{60} alloy, similar experiments were performed in the single-lens focusing column to determine primarily the effects of the unseparated beam on resist exposure. Although the column potential was fixed at 55 keV, the dose could be varied over a very wide range. Line scans 100 μm long were drawn in 1-μm-thick PMMA at scan speeds ranging from 2 μsec/pt to 20 μsec/pt. As before, the development time in 30% MIBK was 60 sec. The resulting developed pattern is shown in Figure 15. The lines on the left side of the pattern were done at lower doses (fast scan speeds), while the lines at the right were done at higher doses. As can be seen, the resist began to polymerize due to the heavy gold dose after about the third line scan. SEM photographs of the lines are shown in Figure 16 through 19. These lines, exposed with a total source current of ~20 μA, resulted in a linewidth at the fastest scan speed of ~4000 Å. This beam size is in good agreement with the gallium ion source. Although the ion current in the single-lens column was not accurately measured, a reasonable estimate indicates a Be^{++} dose of 3 x 10^{12} Be^{++}/cm^2 for Figure 16 and a dose of 6 x 10^{12} Be^{++}/cm^2

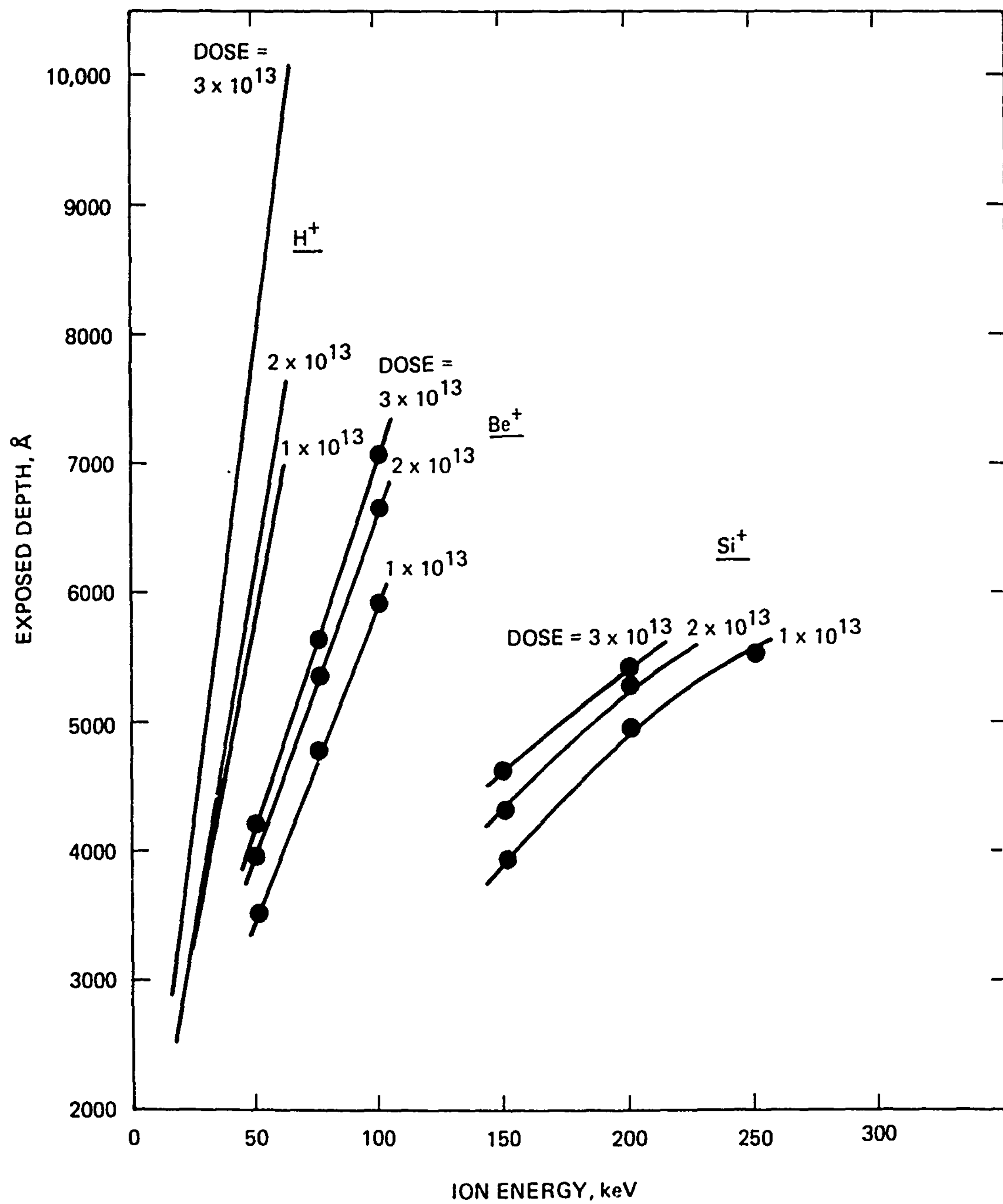

Figure 13. Exposed depth in PMMA for Si^+, Be^+, and H^+ ions as a function of beam energy.

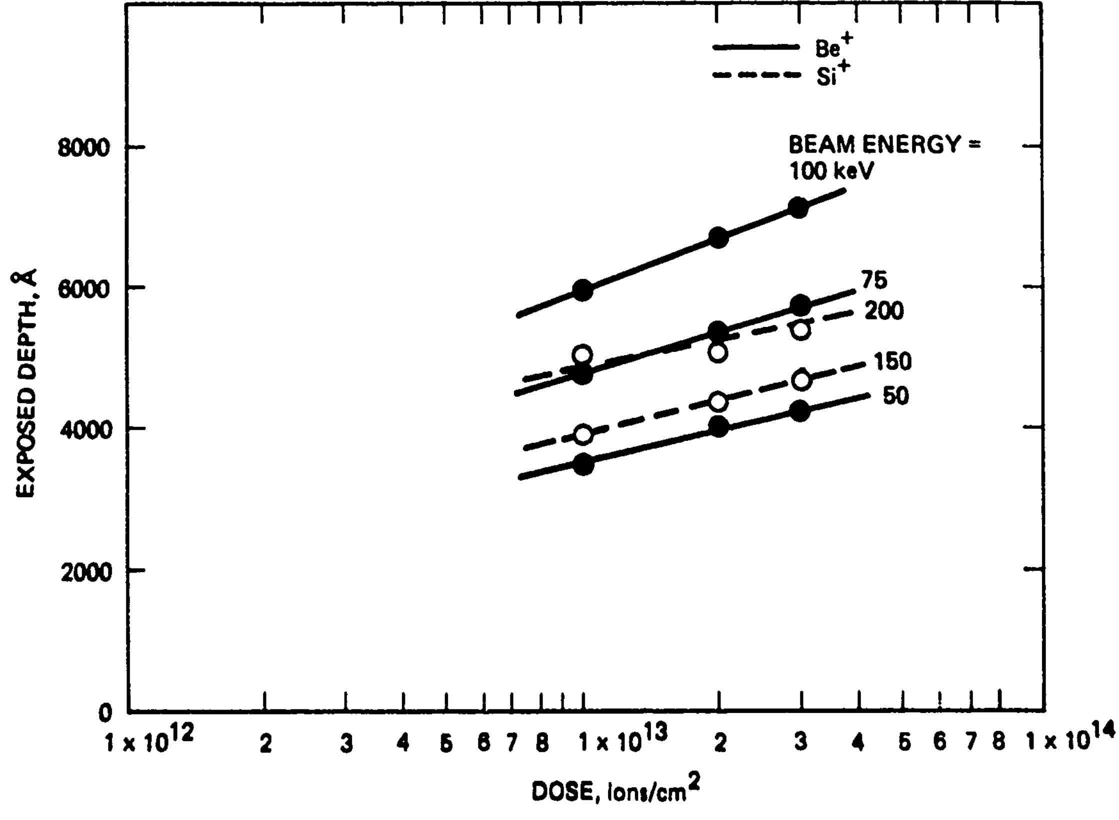

Figure 14. Crossplot of Figure 13 showing exposed depth in PMMA as a function of dose with beam energy as a parameter.

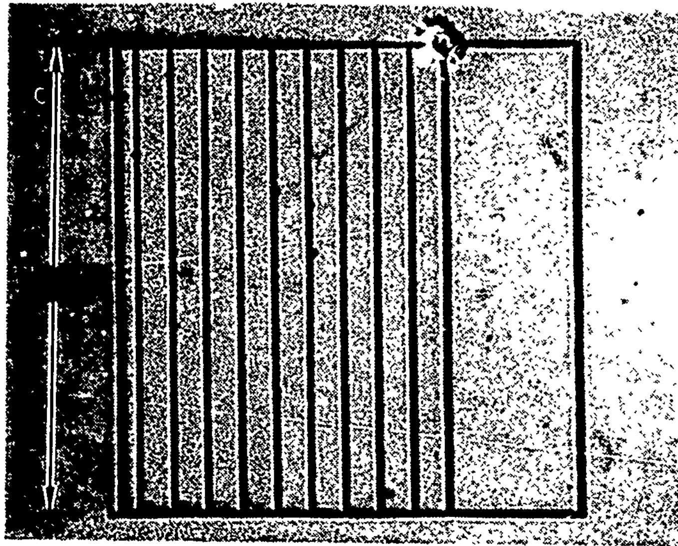

Figure 15. Line pattern in PMMA using the focused Au_{60}-Be_{40} ion beam.

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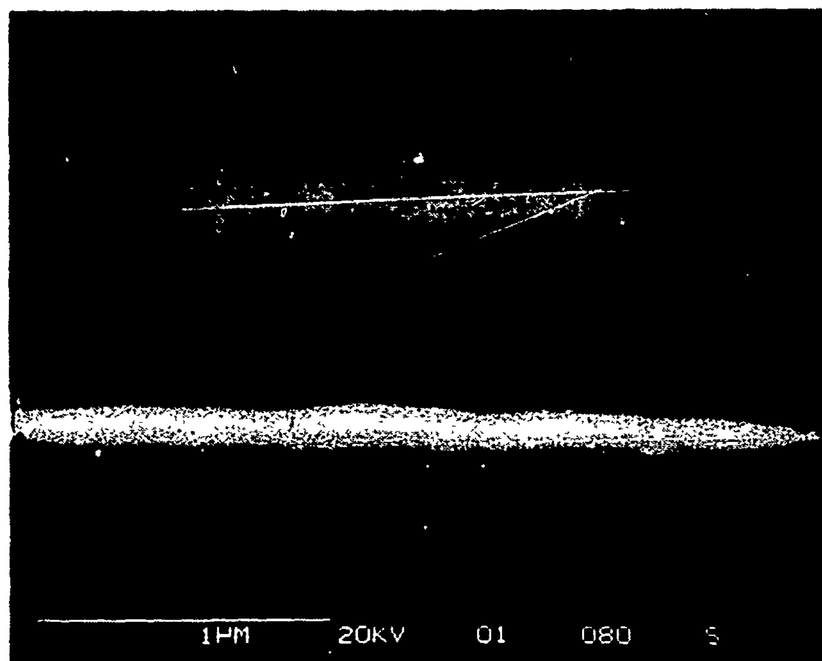

Figure 16. Line scan in PMMA; 2 μsec/pt.

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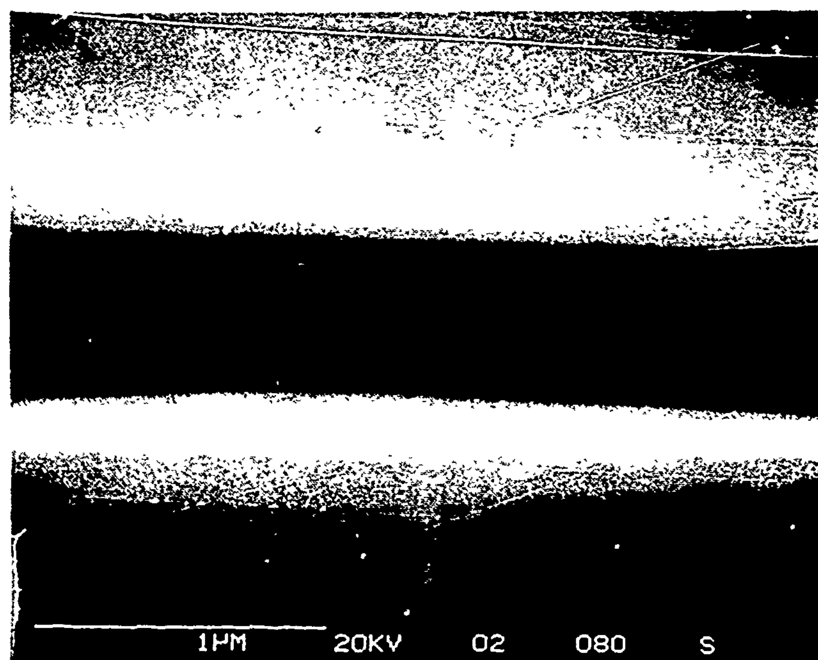

Figure 17. Line scan in PMMA; 4 μsec/pt.

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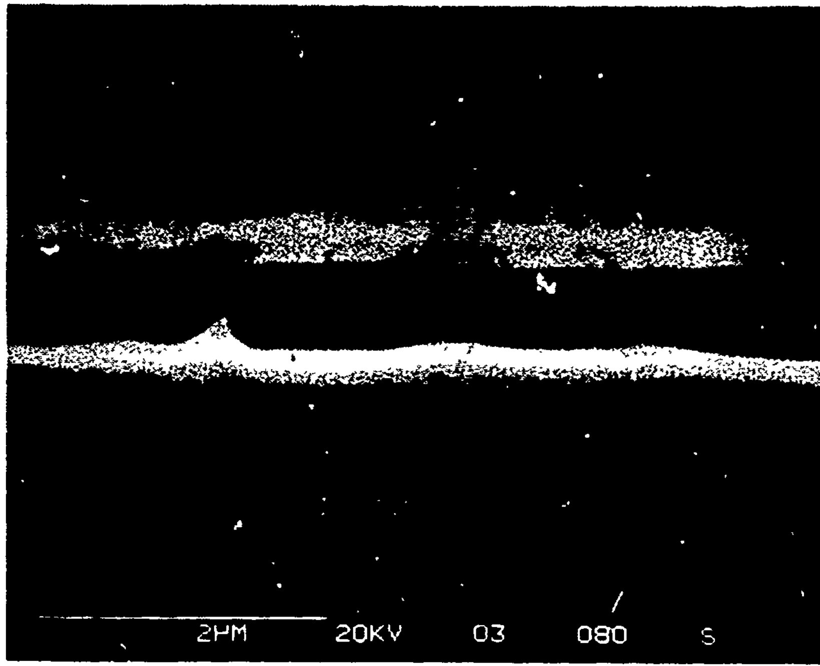

Figure 18. Line scan in PMMA; 6 μsec/pt.

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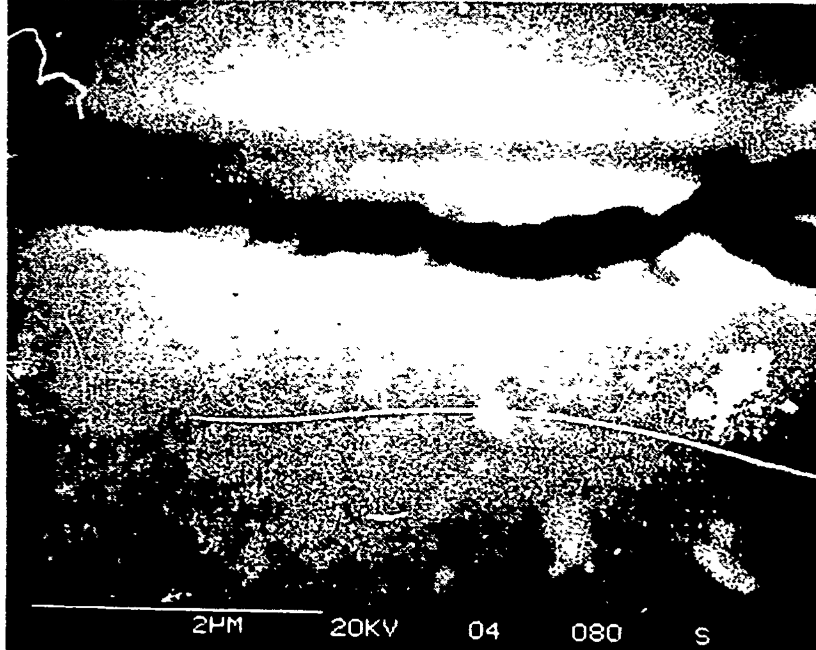

Figure 19. Line scan in PMMA; 8 μsec/pt.

for Figure 17. Figures 18 and 19 show the onset of top-layer polymerization at doses of $\sim 9 \times 10^{12}$ Be^{++}/cm^2 and $\sim 12 \times 10^{12}$ Be^{++}/cm^2, respectively. A preliminary depth evaluation of the low-dose line reveals an exposed channel ~3000 Å deep. SEM techniques are presently being refined to obtain a detailed edge profile. Figure 20 shows a low-dose ($\sim 3 \times 10^{12}$ Be^{++}/cm^2) line exposed with a source current of ~10 μA. The linewidth in this case is ~3000 Å, again in good agreement with the Ga and Au-Si ion sources.

The low-dose requirement for the Au-Be alloy does not present any particular problem since exposed depth can be increased simply by increasing the beam energy. In the case of the Au-Si alloy, however, the maximum dose may require a column potential in excess of 150 kV to obtain an appreciable range (~0.5 μm). Thus, an investigation of other aloys, such as Al-Si or Cu-Si, may be necessary. Resist exposure with the Au-Si alloy is planned for next quarter, and more definite conclusions can be made at that time.

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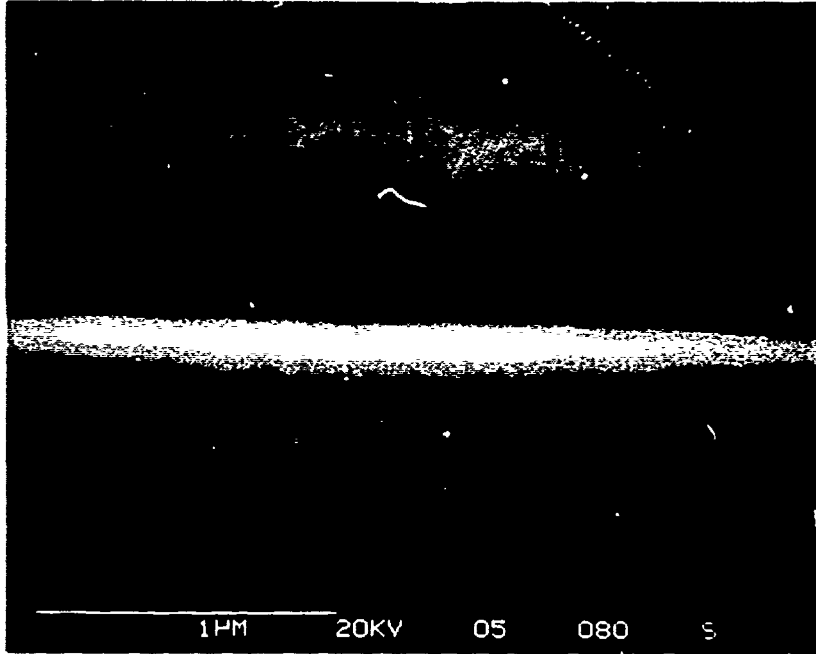

Figure 20. Line scan in PMMA; 2 μsec/pt with I_{source} reduced to ~10 μA.

SECTION 4

CONCLUSIONS

Several ions are potential candidates for resist exposure at 150 keV, including $^{9}Be^{+2}$, $^{12}C^{+2}$, $^{11}B^{+}$, $^{7}Li^{+}$, and $^{28}Si^{+2}$. Of these, beryllium and silicon appear to be the most promising; however, all the ions will receive further consideration. If alloyed materials prove to be unsatisfactory in future tests, lithium appears to be the best choice for a single-ion species if a suitably inert atmosphere can be provided in a practical system configuration.

Resist exposure with both flood and focused ion beams indicates that:

- Mass-separated beryllium and silicon ions appear to have sufficient range in PMMA at energies below 150 keV.
- As expected, exposed linewidths in PMMA are a function of beam energy and dose. There is a definite onset of polymerization of the resist for the Au_{60}-Be_{40} alloy at doses above $\sim 9 \times 10^{12}$ Be^{++}/cm^2.
- For exposures in which polymerization did not occur, the linewidths are in good agreement with previous column operation with gallium. At these lower doses, the exposed depth can be increased by increasing beam energy.
- The column potential for silicon ion exposures at maximum dose (onset of polymerization) may exceed 150 kV. More detailed study is required.
- Exposed linewidths in PMMA are equal to the focused-ion-beam diameter. For example, the beam diameter for these exposures was 3000 to 4000 Å as determined by the lens aperture. By reducing aperture size, the beam diameter will decrease to 1000 to 2000 Å. We expect that the linewidths exposed by the smaller spot will simultaneously decrease to this value, and we will demonstrate this during the next quarter.

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