JIRM 1999; 1(2): 1-6
Penerbit: RCMS

Jurnal Ilmu dan Rekayasa Material
ISSN: 1411-0202

# Boron Liquid Metal Ion Source for Focused Ion Beam System

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**R.H.Rusli**. Boron Liquid Metal Ion Source for Focused Ion Beam System. Jurnal Ilmu dan Rekayasa Material. Agustus 1999; 1(2): 1-6

## Abstract

Boron liquid metal ion sources (LMIS) for focused ion beam system have been developed in the form of binary eutectic alloy of $Pd_{73}B_{27}$. This alloy source was selected as a p-type dopant source. A source life time of more than 120 h has been recorded with different emitter tip radii to test boron ion beam stability. Microstructure examination of the $Pd_{73}B_{27}$ binary alloy proved that boron LMIS instability was cause primarily by the formation of solid precipitates due to a change in alloy stoichiometry. Auger electron spectroscopy (AES) analysis of boron beam deposited on a flat silicon substrate shows rhenium emitter erosion as well as other elements (Fe, Ni and Cr) resulting from extractor sputtering. Greater attention was paid on the metallurgical aspect of LMIS to develop more reliable boron LMIS.

## Abstrak

Sumber ion metal cair boron untuk sistem berkas ion terpusat telah dibuat dengan menggunakan paduan biner "eutectic" $Pd_{73}B_{27}$. Sumber berkas ion dari paduan ini dipilih sebagai sumber dopan jenis p. Masa pakai paduan lebih dari 120 jam telah dicatat dengan percobaan menggunakan berbagai ukuran jari-jari emiter untuk mengukur kestabilan dari berkas ion yang berbasis boron. Penyelidikan terhadap struktur mikro dari paduan biner $Pd_{73}B_{27}$ membuktikan bahwa ketidak stabilan dari sumber ion metal cair berbasis boron disebabkan oleh pembentukan presipitat padatan. Pembentukan presipitat padatan ini akibat dari perubahan "stoichiometry". Analisa dengan menggunakan spektroskopi Auger dengan cara mendipositkan berkas ion ini pada permukaan lempeng silikon yang rata membuktikan bahwa terjadi erosi emiter yang terbuat dari rhenium dan juga elemen lain akibat dari pengaruh "sputtering" ekstraktor. Perhatian pada penelitian ini ditujukan pada aspek metalurgi dari sumber berkas ion metal cair untuk mengembangkan sumber ion metal cair boron yang dapat diandalkan.

## Introduction

Liquid metal ion source (LMIS) in focused ion beam (FIB) system have emerged as a primary source for recent submicron level fabrication of IC devices.[1] In the FIB system, a LMIS is used as a high brightness source for a variety of purposes such as: direct write dopant source, micromachinning, and photomask repair.

There are several LMIS which offer a variety of metal ion species. Of these, boron attracts considerable interest because it is the most common *p*-type dopant for submicron IC fabrication.

To date, although there is a large body of work on LMIS,[2-6] there is a relatively little information published about the metallurgical aspects of LMIS which affect boron ion characteristics such as source lifetime and ion beam stability. The development of reliable boron LMIS requires a basic understanding of the materials problems which affect lifetime, ion beam stability, and current density. In this paper, we report on the metallurgical aspects of $Pd_{73}B_{27}$ LMIS which have been developed at the Center for Integrated Electronics at RPI.

## Materials and Method

Figure 1 shows the experimental set up of a dual carbon filament LMIS in the mass spectrometer chamber. The setup comprises a retractable shutter, filament, and ionizing chamber for residual gas analysis, and a series of lenses to focus the beam into the quadrupole mass spectrometer (QMS). The quadrupole mass spectrometer was calibrated with gallium LMIS before this experiment to insure accuracy of the mass spectrometer response for the PdB source. Figure 2 shows the dual carbon filament liquid metal ion source built and developed at the CIE-RPI laboratory. This type of LMIS incorporated two graphite ribbon filaments instead of one and has been operated more than120 hour with $Pd_{73}B_{27}$ binary eutectic alloy.

Two graphite ribbon filaments are more stable than a single ribbon filament and provide stable liquid metal flows from the reservoir to the emitter tip. The emitter needle is anchored in the upper graphite ribbon filament and cleaning was performed in vacuum to remove oxide on the needle surface before loading with $Pd_{73}B_{27}$ alloy in the form of lump (1-2 $mm^3$). After cleaning the needle, the alloy lump is immediately inserted between the two graphite ribbons at atmospheric conditions, followed by melting in vacuum at $10^{-7}$n Torr to wet the needle. Liquid metal passage from reservoir to the emitter tip occurs by capillary flows through a small aperture hole in the lower graphite ribbon filament.

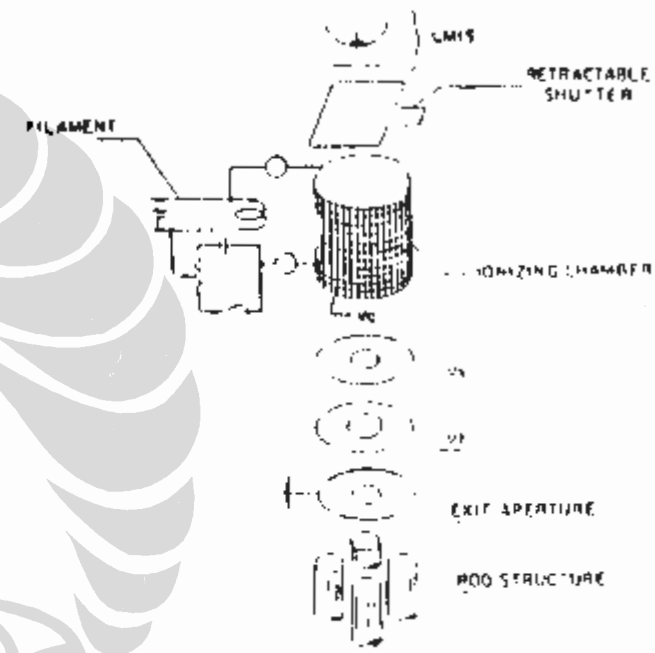


Fig. 1. Liquid metal ion sources experimental setup in the QMS chamber.

Three different emitter radii (2.5, 5.0, and 10 μm) made of rhenium were prepared by mechanical grinding, follow ed bu electrochemical etch (2min) with 5N NaOH. Input heater power in the range of 50-80Watts was require to melt the alloy and maintain the liquid flows to the emitter tip.

## Result and Discussion

### *Mass spectrum & operating behavior*

A typical mass spectrum of $Pd_{73}B_{27}$ LMIS with 2.5 μm radius emitter tip shown in Fig. 3 was obtained from the QMS with a low scanning rate of

0.5 amu/s and a total scanning width of 140 amu on the horizontal scale. The boron isotopic ratio is quite consistent with the natural boron isotopes abundance ratio. The spectrum shows doubly ionized palladium $Pd^{++}$ which is a typical post ionization of singly ionized palladium, along with the molecules of PdB$^{+}$. In Table 1, are tabulated various elements detected by the QMS. The average $^{11}B^{+}$ current recorded (with no attempt to focus the beam) is 0.41 μA which represent 12.13% of the total beam current recorded by QMS. From the tabulated element in Table 1, $(B/Pd)_{beam}$ = 24.13% which is less than $(B/Pd)_{alloy}$. Figure 4 shows $^{11}B^{+}$ current-voltage and total extractor current-characteristics characteristics of $Pd_{73}B_{27}$ alloy with emitter radius of 2.5 μm.

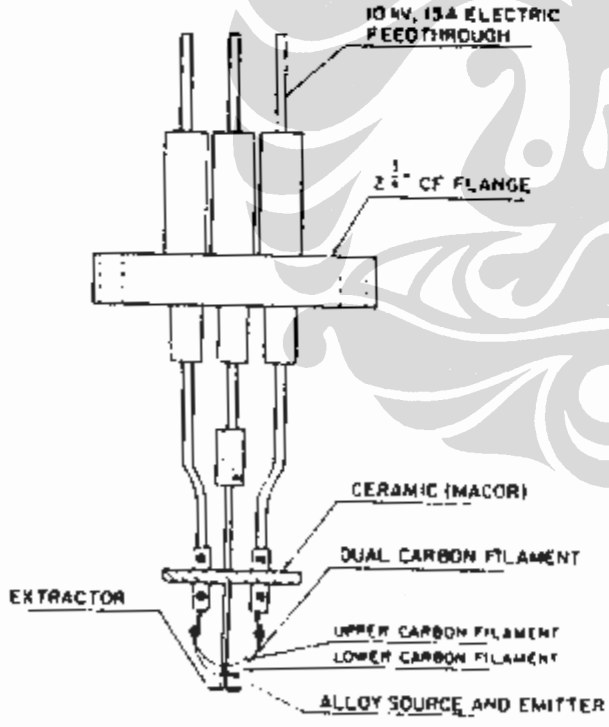


Fig.2. Dual carbon filament liquid metal ion source.

The difference is more significant at higher applied voltage, where the $^{11}B^{+}$ current tends to saturate. Figure 5 shows stability measurement plotted as $^{11}B^{+}$ current vs time for three different emitter radii. For 2.5 μm radius emitter tip (see Fig 5c), the instability is less than 4% and the $^{11}B^{+}$ current drift becomes almost twice as large with an increase of emitter tip radius to 5 and 10μm [see Figs. 5(a) and 5(b)]. The recorded $^{11}B^{+}$ current drift is 8.3% and 7.9% for both 5 and 10μm emitter tip, respectively.

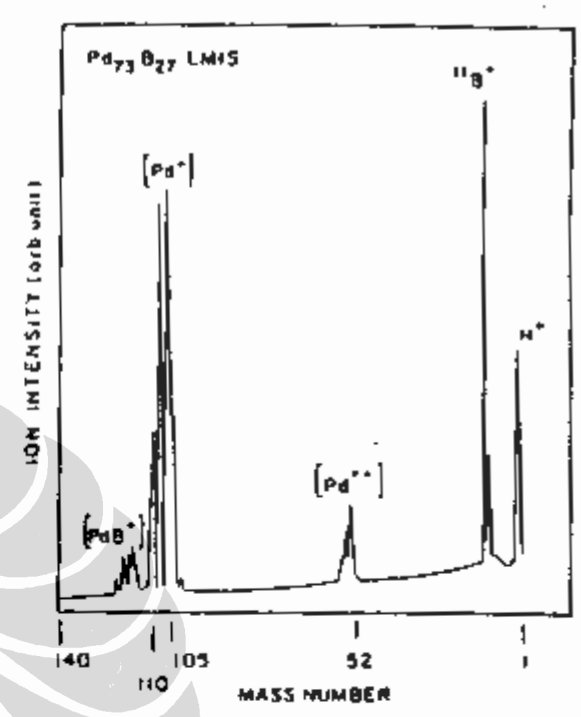


Fig. 3. Mass spectrum of PdB liquid metal ion source.

Table 1. Percentage abundance of various elements in beam at 3.38μA for PdB alloy on Re emitter (tip radius is 2.5μm).

| Elements | Current (A) | % Abundance |
|---|---|---|
| $11_{B}^{+}$ | $4.1 \times 10^{-7}$ | 12.13 |
| $10_{B}^{+}$ | $2.0 \times 10^{-7}$ | 5.91 |
| $104_{Pd}^{++}$ | $1.0 \times 10^{-7}$ | 2.96 |
| $106_{Pd}^{++}$ | $2.3 \times 10^{-7}$ | 6.80 |
| $105_{Pd}^{++}$ | $9 \times 10^{-8}$ | 2.66 |
| $110_{Pd}^{++}$ | $3.0 \times 10^{-8}$ | 0.89 |
| $102_{Pd}^{+}$ | $4.0 \times 10^{-8}$ | 1.18 |
| $104_{Pd}^{+}$ | $4.6 \times 10^{-8}$ | 1.36 |
| $105_{Pd}^{+}$ | $4.5 \times 10^{-7}$ | 13.31 |
| $106_{Pd}^{+}$ | $6.2 \times 10^{-7}$ | 18.34 |
| $108_{Pd}^{+}$ | $6.0 \times 10^{-7}$ | 17.75 |
| $110_{Pd}^{+}$ | $3.0 \times 10^{-7}$ | 8.87 |
| $104_{Pd}{}^{11}{}_{B}^{+}$ | $8.0 \times 10^{-8}$ | 2.37 |
| $106_{Pd}{}^{11}{}_{B}^{+}$ | $7.0 \times 10^{-8}$ | 2.07 |
| $108_{Pd}{}^{11}{}_{B}^{+}$ | $6.0 \times 10^{-8}$ | 1.77 |
| $110_{Pd}{}^{11}{}_{B}^{+}$ | $5.0 \times 10^{-8}$ | 1.48 |

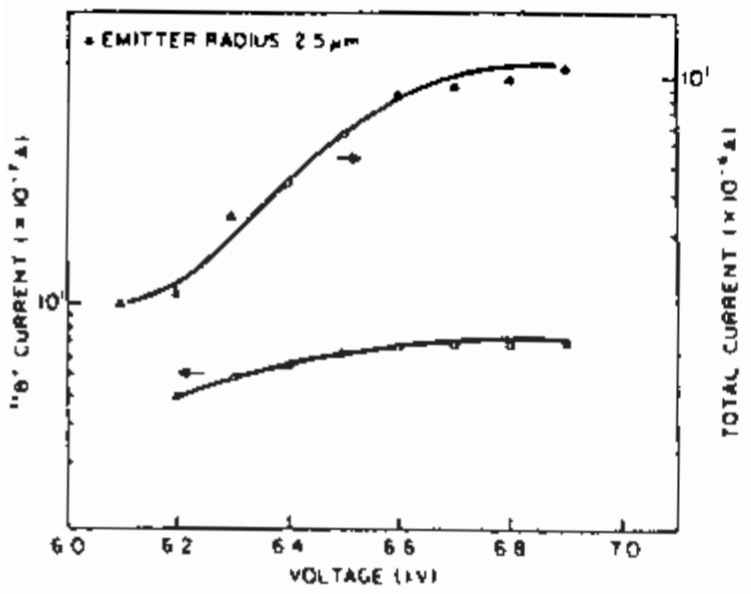


Fig. 4. Current-voltage characteristics of PdB alloy with emitter radius of 2.5μm.

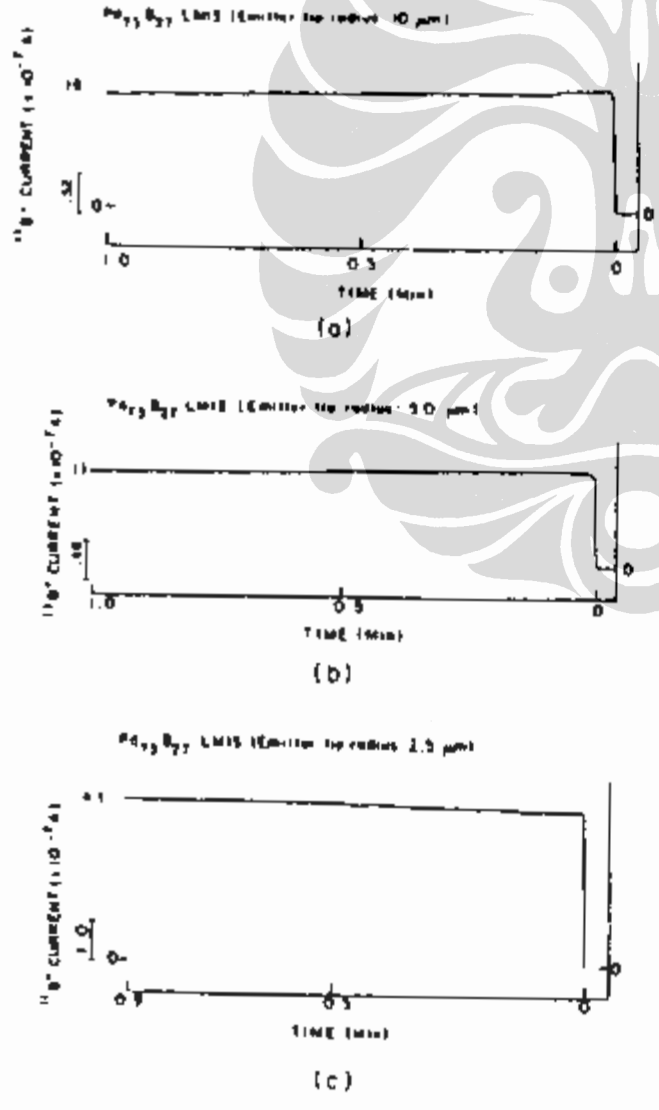


Fig.5. Short tern stability measurement of PdB liquid metal ion source with different emitter tip radius (a) 10μm: (b)5.0μm;(c) 2.5μm.

The $^{11}B^{+}$ current recorded with the same extractor condition also varies with emitter tip radius. For 5 and 10μm emitter radius, the $^{11}B^{+}$ current recorded is 27% and 39% less compare to the $^{11}B^{+}$ current measured with 2.5μm emitter tip

Although short term stability measurement with different emitter tip radii appear to be encouraging with little $^{11}B^{+}$ current fluctuation, nonetheless, long term stability measurement gives result which are quite different than the short term stability measurement. Figure 6 shows a long term test of stability measurements of $^{11}B^{+}$ current with three different emitter radii. The average $^{11}B^{+}$ current drift is less than 4% for 2.5μm emitter tip radius after 120 h test run. For all three emitter tips used in this experiment, large current drift was observed during the first 10 h run. Two other sources with 5.0 and 10μm radius emitter tip did not operate stably and exhibited a shorter lifetime due to mechanical failure of the graphite ribbon fastener.

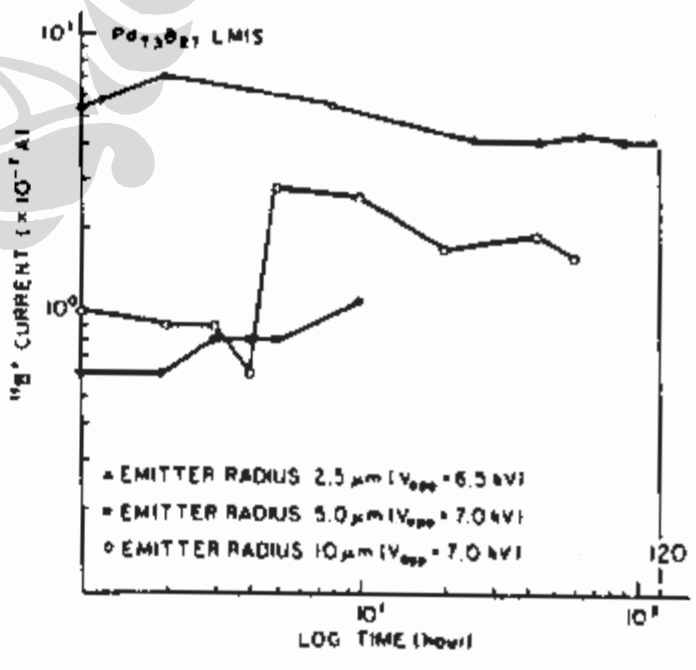


Fig.6. Long term stability measurement of PdB liquid metal ion source with different emitter tip radii.

## *PdB LMIS microstructure*

A typical phase diagram of PdB alloy is shown in Fig.7. The eutectic composition occur at 24.2 at% B and

varies up to 27 at% B.[7,8] Figure 8(a) depicts an electron micrograph of the tip of a wetted 5μm radius of rhenium emitter needle which shows visible solid precipitates of approximately 1μm size. From the phase diagram shown in Fig.7, it is most likely that a slight digression from a single phase eutectic composition during field evaporation shifts the single phase to a two phase region of solid and liquid.

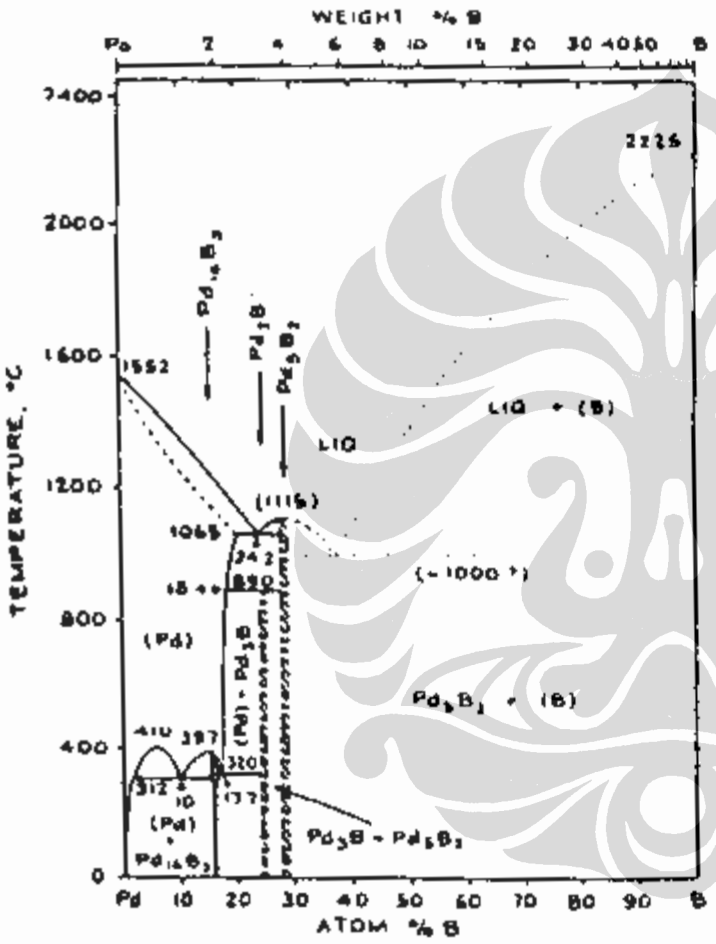


Fig.7. PdB constitution phase diagram (Ref. 10).

Therefore, it is conceivable that the solid precipitates occur because of a change in stoichiometry from a single phase to two phase mixture of solid and liquid. The most probable reason for the change in stoichiometry is a local concentration built up of a segregated Pd-rich alloy from $Pd_3B$ alloy which is left behind during field evaporation. Base on the PdB phase diagram, it is less likely that boron will segregate from $Pd_3B$ to form a B-rich alloy plus $Pd_5B_2$ because this will involve a large change in composition although the $(B/Pd)_{beam}$ is less than $(B/Pd)_{alloy}$, which means more boron is left in the liquid alloy.

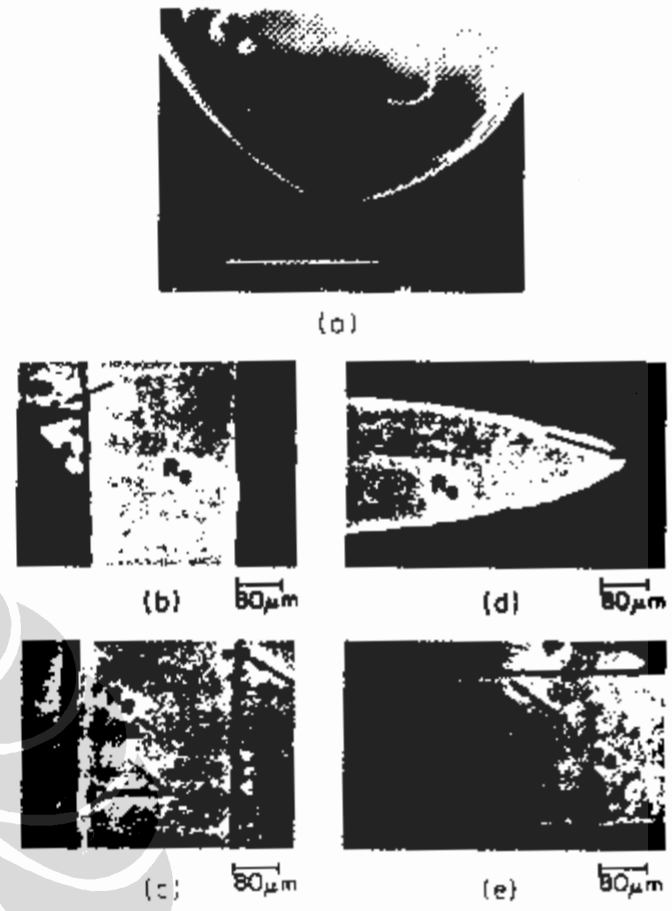


Fig.8. Microstructure of PdB liquid metal ion source; (a) 5 μm tip by SEM; (b) metallograph of a 5μm tip with solid precipitate (c) 5μm tip shows precipitate migration shown by arrow and (d) 10μm tip which shows rounding off the tip (e) 10μm tip with intermetallic precipitates at the interface.

A second possibility to explain the change in alloy stoichiometry is the formation of intermetallic compound precipitates of $Pd_3B$+ $Pd_5B_2$ in the liquid as more boron is left behind and subsequently, the precipitates migrate along the shank to the tip apex of emitter needle as shown in Fig. 8(a-e). These solid phase eventually cause ion beam instability during operation of a focused ion beam system

Figure 9 shows AES analysis of PdB LMIS ion beam deposited in 3 h on a solid silicon surface, which shows rhenium and other elements. Since rhenium ions are not detected by the

QMS, it is more likely that most of the rhenium elements detected by the AES originated from emitter tip erosion and not from rhenium in the liquid alloy. Therefore, although the boron LMIS may last more than 120 h, maintaining beam stability with clean emission free from extractor sputtering elements of Cr, Fe and Ni remain a difficult task. This difficulty will remain in the development of not only boron liquid metal ion source but possible for other liquid metal ion sources as well.

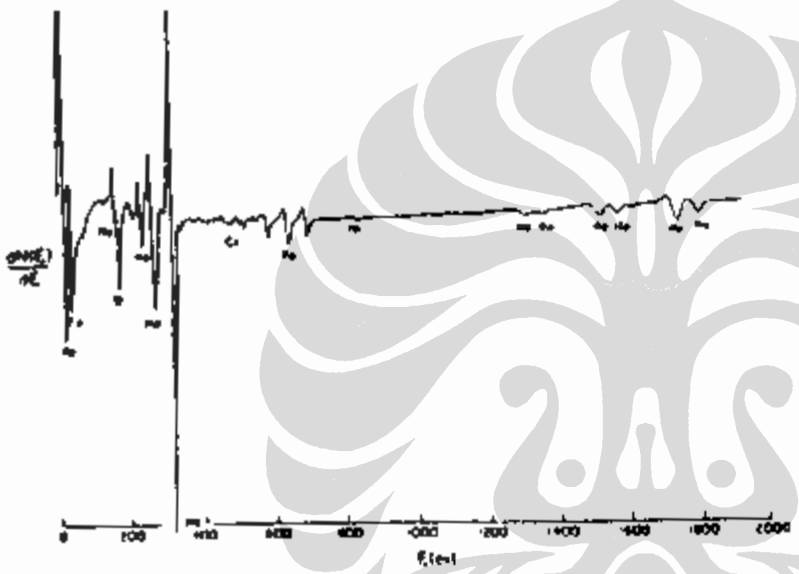

FIG 9. Auger electron spectroscopy of a liquid metal ion sources deposited on a flat solid silicon surface using 10μm tip radius

## Conclusion

PdB binary cutectic alloy is a good candidate for boron LMIS for the FIB application. This criteria stem from a lifetime test of more than 120 h test run. A combination of rhenium as a liquid metal emitter and graphite ribbon as a liquid metal ion source heater is a one possible to produce along lifetime source. The cause of LMIS instability is due to a combination of solid precipitates formation as a result of change in alloy stoichiometry to a palladium rich alloy and the formation of intermetallic compound in the liquid melt. This hinder the flows of alloy to the tip apex, and cause beam instability.

## Acknowledgment

This work is part of the dissertation research done by the author at the center for integrated electronics at Rensselaer Polytechnic Institute, Troy, NY, USA.

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