BNL-112215-2016-CP

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energy semiconductor ion implantation***

**A. I. Hershcovitch¹, V. I. Gushenets², D. N. Seleznev³,
B. A. S. Bugaev², S. Dugin⁴, E. M. Oks², T. V. Kulevoy³,
C. O. Alexeyenko⁴, A. Kozlov³, G. N. Kropachev³,
D. R. P. Kuibeda³, S. Minaev³, A. Vizir², G. Y. Yushkov²**

¹Brookhaven National Laboratory, Upton, NY 11973 USA

²High Current Electronics Institute, Siberian Branch of Russian Academy of
Sciences, Tomsk 634055 Russia

³Institute for Theoretical and Experimental Physics, Moscow 117218 Russia

⁴State Scientific Center of the Russian Federation State Research Institute for
Chemistry and Technology of Organoelement Compounds, Moscow Russia

Presented at the 16th International Conference on Ion Sources
New York Marriott Marquis Hotel, New York, NY
August 23 – 28, 2015

May 2016

Collider-Accelerator Department

Brookhaven National Laboratory

**U.S. Department of Energy
Office of Science, Office of Nuclear Physics**

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Molecular ion sources for low energy semiconductor ion implantation^{a)}

A. Hershcovitch^{1,b)}, V. I. Gushenets², D. N. Seleznev³, A. S. Bugaev², S. Dugin⁴, E. M. Oks², T. V. Kulevoy³, O. Alexeyenko⁴, A. Kozlov³, G.N. Kropachev³, R. P. Kuibeda³, S. Minaev³, A. Vizir², and G. Yu. Yushkov²

¹Brookhaven National Laboratory, Upton, New York 11973, USA

²High Current Electronics Institute, Siberian Branch of Russian Academy of Sciences, Tomsk 634055, Russia

³Institute for Theoretical and Experimental Physics, Moscow 117218, Russia

⁴State Scientific Center of the Russian Federation State Research Institute for Chemistry and Technology of Organoelement Compounds, Moscow, Russia

(Presented XXXXX; received XXXXX; accepted XXXXX; published online XXXXX)

Smaller semiconductors require shallow, low energy ion implantation, resulting space charge effects, which reduced beam currents and production rates. To increase production rates, molecular ions are used. Boron and phosphorous (or arsenic) implantation are needed for P-type and N-type semiconductors respectively. Carborane, which is the most stable molecular boron ion leaves unacceptable carbon residue on extraction grids. A self-cleaning carborane acid compound ($C_4H_{12}B_{10}O_4$) was synthesized and utilized in the ITEP Bernas ion source resulting in large carborane ion output, without carbon residue. Pure gaseous processes are desired to enable rapid switch among ion species. Molecular phosphorous was generated by introducing phosphine in dissociators via $4PH_3 = P_4 + 6H_2$ generated molecular phosphorous in a pure gaseous process, was then injected into the HCEI Calutron-Bernas ion source, from which P_4^+ ion beams were extracted. Results from devices and some additional concepts are described.

I. INTRODUCTION

One of the last remaining frontiers of the semiconductor industry, as well as a major research and development thrust for the semiconductor ion implantation industry, is low energy ion implantation¹. Since the invention of the transistor, the trend has been to miniaturize semiconductor devices. As devices get smaller, shallower ion implantation for semiconductor manufacturing is needed. Consequently, lower energy ion beams are needed. However, low energy ion beam have limited intensity (current), due to space charge blow-up as the Child Law limit is exceeded. Low intensity ion beams result in low production rates. Therefore, low energy ion implanters have low production rates. Consequently, increasing the current of pure, low energy ion beams is of paramount importance to the semiconductor industry nowadays. Ion beams are usually extracted from ion sources, in which ions are generated by electrical discharge of gases or vapors. At a given extraction energy, ion current is limited by the space charge of the ion beam known as the Child law. Neutralizing plasmas, utilized in today's implanters, to reduce space charge offer only a partial solution and often result in implanting undesirable impurities.

Modern semiconductor technology requires an increase in the efficiency and reliability of highly integrated circuits (ICs), enhancement of their functional capabilities, and a decrease in the cost of IC elements and structures. A technique to do this is to increase the integrated circuit density by decreasing the size of IC elements and increasing their number per unit crystal area, which simultaneously decreases their depth. These tasks inspire continuous improvement and development of technological equipment for IC manufacturing. Decreasing the modification depth necessitates a decrease in dopant ion energy; hence the need for shallow implantation and the resultant space charge issues.

Although most implanters still rely on xenon plasma¹ for neutralization, a better approach for mitigating the space charge problem is to utilize molecular ion beams (or clusters terminology used by the semiconductor) for implantation.

Boron and phosphorous clusters have been used by the semiconductor ion implantation industry. Much of the development of boron clusters ion sources has been pioneered at SemEquip lead by Tom Horsky² in generating decaborane ($B_{10}H_{14}$) and octadecaborane ($B_{18}H_{22}$) ion beams; though molecular boron compounds were invented in the Soviet Union as rocket fuel. Similarly, tetratomic phosphorus (P_4) and its ions are of interest as prototypes of small clusters. Horsky³ at SemEquip also generated molecular phosphorous ion beams. Both boron and

^{a)}Invited talk paper, published as part of the Proceedings of the 16th International Conference on Ion Sources, New York City, NY, USA August, 2015.

^{b)}Electronic mail: hershcovitch@bnl.gov.

phosphorous clusters are very promising for high-dose very-low-energy ion implantation in semiconductor industry.

The use of clusters (with n atoms) is promising because at a given accelerating voltage and at the same ion beam current density, for the cluster ions implanted dose rate increases in n^2 times compared to that for monatomic ions³. This may be connected to lower implanted energy per cluster atom and shorter the mean path of the ions in a target. The use of polyatomic molecules for implantation provides, along with the increase in beam perveance and implanted dose, the following significant advantages:

- 1) Eliminate the so-called energy contamination and high-energy tail characteristic of low-energy implantation with a retarding field;

- 2) Greatly improves the quality of a low-energy ion beam (decreases its angular expansion and ensures better angular and spatial distributions) at a target; and

- 3) Allow using the existing doping technology for shallow p-n junctions.

Molecular ion generation that is the subject of this paper has advantages over currently used techniques: boron generation is based on carborane ($C_2B_{10}H_{12}$) that is the most stable of multi-boron molecules that does not contain any elements, which have harmful effect on implanted wafers (far more stable than decaborane or octadecaborane); and, phosphorous molecules are generated via a pure gaseous process. But, carborane leaves carbon residue. Therefore, m- and o- Carborane-1,7-dicarboxylic acid ($C_4H_{12}B_{10}O_4$) were synthesized to self-clean the carbon residue.

II. ADVANTAGES OF NOVEL MOLECULAR ION TECHNOLOGIES

While carborane is the most stable of boron molecules useful for semiconductor ion implantation, its present use is very limited due to the carbon residue issue, which requires ion source cleaning. The m- and o- Carborane-1,7-dicarboxylic acid ($C_4H_{12}B_{10}O_4$) have shown to match and even exceed extracted carborane ion from the ITEP Bernas ion source, while leaving basically no carbon residue. Carborane-1,7-dicarboxylic acid ($C_4H_{12}B_{10}O_4$) has three isomers m, o, and p. Both m and o isomers have shown to generate practically results in terms of clean ion sources and carborane outputs, except for 30°C difference in optimal oven temperature. No experiments were performed with p-Carborane-1,7-dicarboxylic acid ($C_4H_{12}B_{10}O_4$), since the p isomer is very hard and expensive to generate. Hence the p-isomers are of no practical use, and most likely of no commercial value.

For implanting phosphorous in wafers, the working plasma-forming medium in ion implanters is phosphorus vapors produced by evaporation of red phosphorus. In its vaporous state, phosphorus P_4 represents the most stable molecular compound. In the discharge chamber of an ion source under certain experimental conditions, the percentage of polyatomic phosphorus ions (P_2 , P_3 , P_4) in an ion beam can reach 80 %^{2,4}.

The use of red phosphorus as a P_4 molecular vapor source involves a series of problems associated with multiphase nature of red phosphorus, its evaporation rate instability, thermodynamic instability (a possibility to transform from one allotropic state to another), and low evaporation kinetics. The foregoing factors considerably lengthen the time during which equilibrium vapor pressure is established over the heated phosphorus surface and result in unstable vapor supply to the discharge chamber of an ion source. And, equally important for the ion implantation industry is that the use of **ovens** is **time consuming** when switching among implantation species; therefore, many manufacturers implant P^+ even for shallow implantations.

An alternative source of phosphorus as a plasma-forming medium is phosphine – a gaseous compound of phosphorus with hydrogen (PH_3) under normal conditions. In discharge systems of ion sources, phosphine is used mainly to obtain singly charged monatomic phosphorus ions. In addition to phosphorus ions, the beam contains ions of phosphorus compounds with hydrogen: PH^+ , PH_3^+ . The total current of PH^+ , PH_3^+ ions is rather high and can reach 25–30% of the total beam current⁵. The use of the gaseous compound allows control and stabilization of the gas flow rate to the discharge chamber with special valves operating at a speed much higher than the temperature variation rate of red phosphorus.

At a temperature of 500°C, phosphine begins to decompose into phosphorus and hydrogen⁶. The decomposition is used to obtain neutral particle beams for molecular epitaxy. For the generation of molecular beams, phosphine is passed through a dissociator. The peculiarity of dissociators used in sources of molecular beams is that the devices are optimized to obtain phosphorus dimers and also arsenic and antimony dimers^{7,8}. This is attained due to appropriate operating conditions and materials used in the design of the dissociator active zone.

Research results from modified ion sources operating with m- and o- Carborane-1,7-dicarboxylic acid ($C_4H_{12}B_{10}O_4$) in the ITEP Bernas ion source and with phosphine as a molecular phosphorus vapor fed into the HCEI Calutron Bernas ion source are presented next.

III. BORON AND PHOSPHOROUS MOLECULAR ION BEAM GENERATION

Molecular ion beams were extracted from ion sources at ITEP (the Bernas) and at HCEI (Calutron Bernas).

A. Molecular boron

As mentioned earlier, carborane ($C_2B_{10}H_{12}$) leaves carbon residue, which is unacceptable for ion implanters when carbon is deposited on grids and creates “shadows” on wafers. Therefore, a number of compounds with potentially self-cleaning properties were synthesized and experimented with. All compounds were carborane molecules, to which self-cleaning elements were attached. During ionization, these elements become loose and remove carbon. Three possible processes of chamber

cleaning during the operation, based on reaction with carbon, were tested. First was fluorine, which is used by the ion implantation industry (usually between ion source operations). Fluorinated carborane ($C_2B_{10}H_{11}-F$), in which one hydrogen atom was replaced by a fluorine. Second cleaning mechanism was based on so called Boudouard reaction where source surface is cleaned from carbon by CO_2 produced in the discharge chamber as result of $C_4H_{12}B_{10}O_4$ or $C_3H_{12}B_{10}O_2$ (o-carborane 1-carboxylic acid) molecular fragmentation by the reaction $CO_2 + C \rightleftharpoons 2CO$, which goes from left to right at temperatures higher than $400^\circ C$. Pure oxygen, from molecular fragmentation, can clean surfaces by forming CO_2 and CO that are pumped out. The difference between $C_3H_{12}B_{10}O_2$ and $C_4H_{12}B_{10}O_4$ is that the first contains one C-O-O-H acid molecule, while the latter is bi-acid.

All experiments were performed using the ITEP Bernas ion source, which is described fully elsewhere⁹; first in a water-cooled copper chamber, and later in cooled and uncooled molybdenum discharge chambers. To measure charge-state distributions, we used a mass analyzer with a magnet bending radius of 0.35 m and a bending angle of 60 degrees. No structural modifications were needed for carborane operation. Nevertheless different operating parameters were needed for each compound. Oven and transfer line (to discharge chamber) temperatures were very critical; they ranged from 140-400°C in the transfer line, and 200-500°C in the oven depending on the compound. Temperature control required care, since transfer line heating affected the oven temperature and vice versa; uncooled chamber operation had only slight affect. The transfer line was heated first.

The ITEP Bernas is a small ion source with an extraction slit of $1 \times 20 \text{ mm}^2$. Common operating set parameters (to enable comparison between compounds) were extraction Voltage of 10 kV and arc Voltage of 250 Volt. In most operations, the pressure (measured outside the chamber) was 6×10^{-5} Torr. Ion source operation was 4 hours steady state.

Ion source operation with fluorinated carborane ($C_2B_{10}H_{11}-F$) showed some reduction in carbon deposition, suggesting that many hydrogen atoms must be replaced with fluorine; extremely difficult to synthesize. O-carborane 1-carboxylic acid ($C_3H_{12}B_{10}O_2$) cleaned much of the residue, while after 4 hours of operation in m- or o-carborane-1,7-dicarboxylic acid ($C_4H_{12}B_{10}O_4$) ion source chamber and extractors were completely clean (no isomer effect was observed). Next comparison is made between carborane 1-carboxylic acid (to be referred to as acid or carborane acid) and pure carborane operation.

FIG. 1. ITEP Bernas ion source chamber photos after carborane (left) and acid (right) operations of 4 hours steady state.

Figure 1 shows the water-cooled copper chamber of the ITEP Bernas ion source after 4 hours of steady state operation with pure carborane and with acid. It's obvious from figure 1 that carborane-1,7-dicarboxylic acid does self-clean the ion source. Next a molybdenum chamber was fabricated for the ITEP Bernas ion source, which was then operated with and without cooling. Pure carborane operation resulted in debris as shown in figure 1 after 4 hours of operation. Figure 2 displays photos of the ITEP Bernas molybdenum discharge chamber and extractor, which remain very clean without any carbon residue after hours of operation in o- carborane-1,7-dicarboxylic acid.

FIG. 2. Photos of ITEP Bernas molybdenum chamber (left) and extractor (right) after 4 hours of steady state operation.

Surprisingly, carborane current extracted from the ITEP Bernas ion source during o-carborane-1,7-dicarboxylic acid operation was somewhat higher than carborane current extracted during pure o-carborane operation as it can be seen from figure 3. Bottom curve of

Figure 3 o-isomer comparison between carborane spectrum (lower curve) and o-carborane-1,7-dicarboxylic acid (upper curve).

that figure displays extracted molecular ion spectrum during carborane operation, on which extract molecular ion spectrum during o-carborane-1,7-dicarboxylic acid operation is superimposed. **Total current extracted during acid operation was 0.3 mA, while during pure carborane operation it was 0.1 mA.** The reason for a higher carborane output during o-carborane-1,7-

dicarboxylic acid operation than pure carborane, is most likely due to higher discharge current 20 mA versus 16 mA (arc Voltage, extraction Voltage and pressure were identical in both cases), and/or higher plasma density in the ion source chamber; most likely the two parameters are interrelated.

Extracted spectrum of both carborane and acid beams were very stable during the 4 hour operation; care was taken to stabilize the oven, transfer line and ion source, which contributed to operation stability. Operation was reproducible once proper conditions were reached. Possibly optimal temperatures lead to best surface conditions. In figure 3, the first large sharp peak of the acid spectrum is CO, while the next peak is CO₂. For ion implantation the carborane peak at 144 is to be utilized. Indeed higher masses than this peak are to the formation of bi- o-carborane-1,7-dicarboxylic acid and its fragments.

At chamber temperatures higher than 250°C, some traces of carbon residue were observed after 4 hours of acid operation. It could be a concern for industrial ion sources that operate at even higher temperatures continuously for 168 hours. Problem was solved by injecting traces of atomic oxygen, which lead to experiments where oxygen was injected in pure carborane operation, which yielded results similar to acid. Given these, it opens the possibility of a much simpler solution by co-injection of oxygen. Injecting atomic oxygen directly on carborane ion sources extraction grids and metal faceplates has added benefits: ionized oxygen will not be extracted at the expense of implantation species current, and it won't erode graphite sources. This can be done by flowing O₂ into an elliptical cross section dissociator and generating an atomic oxygen beam, similar to the way it was done for atomic hydrogen¹⁰.

Finally co-injecting CO₂ with carborane into the discharge chamber did have any self-cleaning affect. Therefore, self-cleaning is likely due to oxygen released from the carborane acid. Nevertheless, the Boudouard reaction cannot be excluded at higher temperatures, since none of the experiment exceeded 400°C.

B. Molecular phosphorous

For molecular phosphorous generation, HCEI Calutron-Bernas ion source had to be modified, especially by the addition of a dissociator. Schematic of the modified Calutron-Bernas ion source is shown in figure 4.

FIG. 4. Schematic of the ion source (1-7) and dissociator (8-11). 1 – discharge chamber, 2 – directly heated cathode, 3 – shield, 4 –

anecathode, 5 – supressor, 6 – accelerating electrode, 7 – vapor line, 8 – case, 9 – thermal shield, 10 – quartz tube, 11 – spiral heater.

The ion source uses a hot-cathode discharge in a magnetic field. The discharge chamber 1, which serves as the discharge anode, is made of molybdenum. At the faces of the discharge chamber, a U-shaped directly heated cathode 2 and an electron collector 4 are located. Connecting the collector to the anode results in single pass of electrons emitted from the cathode discharge. This mode is most suitable for generating molecular ions³⁻⁵.

Phosphine is injected into a dissociator, which does not contain catalysts. The dissociator active zone consists of a spiral heater 11 and a quartz tube 10. The phosphine flow rate is controlled by a leak valve. Dissociation products move through a vapor line 7 to the ion source discharge chamber. Thermal contact dissociation on the heater and recombinations on the quartz and in the transfer line for formation of P₄ molecules at temperatures lower than 1000 °C has higher fraction than other reactions⁷ without any catalysts.

The ion source has an extraction slit of dimensions 1 × 40 mm². The ion-optical system is a standard two-electrode system. The ion beam accelerated to a voltage of 15 kV is separated by a sector magnet. The beam is measured using the collector with a slit diaphragm. The current in the separator winding has a saw-tooth shape with a rise time of up to 8 s. The collector signal and the Hall probe signal, which is proportional to the magnetic field in the separator gap, are transmitted simultaneously to the inputs of a Tektronix storage oscilloscope. Thus, the mass-charge state of the ion beam is recorded in a single stroke of the sawtooth current in the separator magnet coil.

Figure 5 shows the ion beam constitution depending on the temperature of the spiral heater (11) in the dissociator active zone. As the spiral temperature

FIG. 5. Ion beam component as function of dependence on dissociator active zone heater temperature.

comes close to 700 °C, the PH and PH₃ components of the ion beam disappear almost completely, the ion current of phosphorus dimers doubles, and the current of P₄ ions increases more than 4 times. The temperature of the dissociator heater was not measured; it was estimated from the resistance of the heater wire. As can be seen from the

ion beam spectrum, phosphine contains diphosphine P_2H_4 which increases the amount of phosphorus dimers in the dissociation products because the most probable reaction of phosphine decomposition is $P_2H_4 = P_2 + 2H_2$. Absence of hydrogen ions in the beam is due to its deflection in extraction area by fringing H field of the discharge unit.

Mass and charge state of ion beam component dependence phosphine flow rate, discharge current, and discharge operating voltage were studied to optimize P_4^+ ion current. Optimal P_4^+ ion current is achieved operating pressure range of 6.5×10^{-5} to 9×10^{-5} Torr.

FIG. 6. Optimal mass and charge state of the ion beam.

As the voltage is increased from 200 to 570 V, the P_4^+ ion current increases near monotonically, whereas other ion beam components peak at a voltage of about 400 V; discharge currents were 100–200 mA. As the discharge current is increased, the amplitudes of all beam components increase monotonically, except for the P_4^+ ion current, which peaks at discharge currents of 140–160 mA and then decreases. Figure 6 shows an oscillogram of the ion beam spectrum at a maximum total current of 3 mA, of which the P_4 ion current is over 30% of the total beam current. No hydrogen ions are observed in the beam.

IV. ADDITIONAL LOW ENERGY ION IMPLANTATION TECHNOLOGIES

Along with molecular ions two novel low energy ion implantation techniques were developed: gasless plasmaless deceleration and pure boron plasma immersion.

A. Gasless plasmaless deceleration

Presently in ion implanter¹ beams are extracted from ion sources and accelerated at high energy to maximize current. Deceleration to low energy utilizes neutralizing plasmas¹ to reduce space charge, which often result in implanting undesirable impurities.

A novel LEBT is being developed to provide better bending magnet molecular beam transmission facilitate beam slowing down. An electrostatic focusing system with long focus length was fabricated and had improved beam transmission through a separating magnet. After the

bending magnet, an electrostatic undulator is being developed as a transport channel for total carborane beam deceleration and transmission to the target. Simulation of the combined system showed that a 0.75 mA carborane ($C_2B_{10}H_{12}^+$) ion beam was successfully extracted at 10 KV and decelerated down to 2 KV without space charge “blow-up” (beam expansion).

B. Pure boron plasma

Pure boron magnetron discharge with thermally isolated boron target in self-sputtering mode was achieved at HCEI utilizing two methods to heat a 2” boron cathode to at least 500°C: heating the cathode with halogen lamps and a reflector; warming the boron in an argon discharge until the cathode can sustain the discharge in pure boron.

Boron was extracted at 15 KV from the plasma. Analysis was performed: all the boron ions are single charged with boron ion fraction greater than 99%. And, boron ion implantation into silicon wafer samples was performed. Original surface resistance of wafers was in the range of megohms. After implantation, the resistance predictably reduced to several kilohms, which indirectly confirms that there is no boron surface deposition.

V. DISCUSSION

Presented results are self-explanatory. It is important to note that self-cleaning molecular carborane operation is a pioneering achievement. And, prior attempts¹¹ to dissociate phosphine reached a P_4 fraction of only 10^{-4} . In self-cleaning acid carborane and molecular phosphorous operation no other gases were introduced.

The work was supported in part by Plasma Sources LTD, Tomsk, Russia, under a grant from the Skolkovo Foundation, and in part under Contract No. DE-AC02-98CH1-886 with the US Department of Energy.

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