# Boron Liquid Metal Alloy Ion Sources for Special FIB Applications

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# Boron Liquid Metal Alloy Ion Sources For Special FIB Applications

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Focused Ion Beam (FIB) processing has been established as a well-suited and promising technique in R&D in nearly all fields of nanotechnology for patterning and prototyping on the µm-scale and below. Liquid Metal Alloy Ion Sources (LMAIS) represent an alternative to expand the FIB application fields beside all other source concepts. The need of light elements like B was investigated using various alloys. A promising solution was found in a $Co_{31}Nd_{64}B_5$ based LMAIS which should be introduced in more detail. Beside Co ions as a ferromagnetic element and the rare earth element Nd especially B is interesting for special FIB applications with a best obtained resolution of about 30 nm so far.

## I. INTRODUCTION

As a key technique Focused Ion Beams (FIB) devices are well established in nanotechnology for local surface modification, doping, prototyping as well as for ion beam analysis. The main component of such a FIB system is the ion source and the available ion species therein [1]. Most of the instruments at present work with a Ga liquid metal ion source (Ga-LMIS), but the aspiration for other ion species is still increasing [2]. A very distinguished element of interest is boron, one of the lightest elements in the periodic table and well established in microelectronics for p-type doping in silicon by implantation or diffusion [3]. There is a long term interest and a lot of efforts were done for the application of boron in a LMAIS for local material modification by FIB in order to avoid B-broad beam implantation and lithography steps. Boron has two stable isotopes with a mass of 10 u (19.9% natural

abundance) and 11 u (80.1% natural abundance). The melting point of B amounts to $T_{melt}$ = 2080°C with a vapor pressure of about $10^{-8}$ mbar at 1282°C. For the operation in an ion source a considerable lower temperature is needed which can be obtained by a suitable alloying. Pd containing alloys like $B_{10}As_{10}Ni_{40}Pd_{40}$ and $B_{20}Ni_{40}Pd_{40}$ with a melting point of $T_{melt}$ = 650°C [4], $Ni_{45}B_{45}Si_{10}$ with $T_{melt}$ = 950°C [5] as well as the platinum alloy $Pt_{72}B_{28}$ with $T_{melt}$ = 790°C [6, 9] were investigated and tested. A more complete list of B-LMAIS with corresponding references can be found in Ref. [2]. Unfortunately, only a limited extent about details of operation, lifetime, emission currents or instabilities is published in these references. Finally, there is no such B-containing ion source at present available on the market for FIB applications.

In this contribution different LMAIS were prepared, tested and characterized due to their ability for local boron applications in FIB technology.

# II. EXPERIMENTAL

Hairpin emitters with different reservoir geometries were fabricated from 250 µm metallic wires and spot welded on a filament. The tip was sharpened electrochemically for W and Re and accordingly mechanically for Ta to a final radius of (4 ± 1) µm. After heating to 1200°C in UHV (< $10^{-7}$ mbar) the emitters were wetted in a crucible with the chosen alloys, see table 1. A slowly heating of the crucible as well as of the emitter is necessary to obtain a homogeneous temperature distribution in the source reservoir. The filament and tip material was chosen with respect to the wetting behavior and chemical compatibility. In the same geometry the emission was first tested against the cold crucible to minimize the contamination by back-sputtered material. The handling of the emitter for introducing it into a cartridge etc. has to be done at ambient pressure very quickly or in Ar atmosphere. This includes the transportation of B containing LMAIS in Ar filled containers due to cleanliness requirements.

TABLE 1: Source materials, melting temperature, emitter tip material and content of B.

| Source material | $T_{melt}$ (°C) | Emitter | Content of Boron | |
|---|---|---|---|---|
| $Au_{77}Si_{18}B_5$ | 370 | Ta, W | $^{11}B^+/Au^+$ | $< 10^{-5}$ |
| $Au_{70}Ge_{25}B_5$ | 370 | Ta, W | $^{11}B^+/Au^+$ | $6x10^{-4}$ |
| $Au_{68}Ge_{22}Ni_5B_5$ | 370 | Re | $^{11}B^+/Au^+$ | $4x10^{-3}$ |
| $Co_{31}Nd_{64}B_5$ | 650 | Ta, Re, W | $^{11}B^+/Co^+$ | 0.1 |
| $Ni_{40}B_{60}$ | 1032 | W | No B emission | |

All loaded and wetted emitters showed a good emission performance and were than introduced in a cartridge to characterize them in an analytic test-FIB described in [7]. Measurements of the *I-V* curve, the mass spectrum, current stability, live time and the energy distribution for selected ion species depending on the emission current and were

carried out for those having remarkable boron content in the mass spectrum. The ratio of boron with respect to the main peak intensity is also presented in table 1. The most promising candidate $Co_{31}Nd_{64}B_5$ LMAIS was finally installed and investigated in detail in a mass-separated FIB/SEM system (VELION Raith GmbH) [8].

# II. RESULTS AND DISCUSSION

## A. *$Au_{77}Si_{18}B_5$ and $Au_{70}Ge_{25}B_5$*

As a first attempt boron was added to classical metallic glasses (AuSi, AuGe) and tested on different needle materials. Due to the low content of boron in the alloy the sources could be operated at temperatures slightly above the eutectic temperature of 365°C [9]. This low temperature is favorable due to out diffusion and evaporation of B. The relative intensity of the B – peaks are about 3 orders of magnitude smaller than Au, shown in Fig. 1 for AuGeB. In the case of AuSiB the relation was still much more inferior so that these sources could not be used for practical applications.

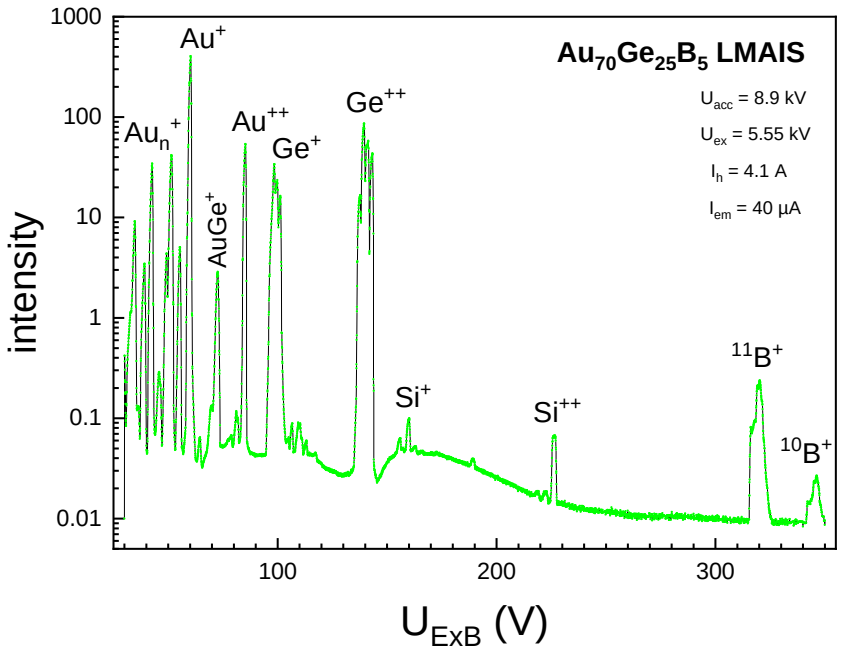



Figure 1: Mass spectrum of an $Au_{70}Ge_{25}B_5$ LMAIS. The current $I_{SEV}$ in the test-FIB [7] was measured with a secondary electron multiplier and is given in arbitrary units.

## B. *$Au_{68}Ge_{22}Ni_5B_5$*

In a second approach a small content of Ni was added to AuGeB to form an $Au_{68}Ge_{22}Ni_5B_5$ alloy. From previous investigations it is known that B and Ni can work in a $Ni_{40}B_{60}$ LMAIS but it was used on a carbon tip at about 1030°C [10]. This new composition was chosen to lower the operation temperature and also to use a standard hair pin emitter with a metallic

tip, Re in our case. A mass spectrum, obtained in the test-FIB [7] is shown in Fig. 2. Main operation parameters are given in the inset. The large number of lines in the spectrum due to isotopes and compound ions makes high resolution FIB application difficult.

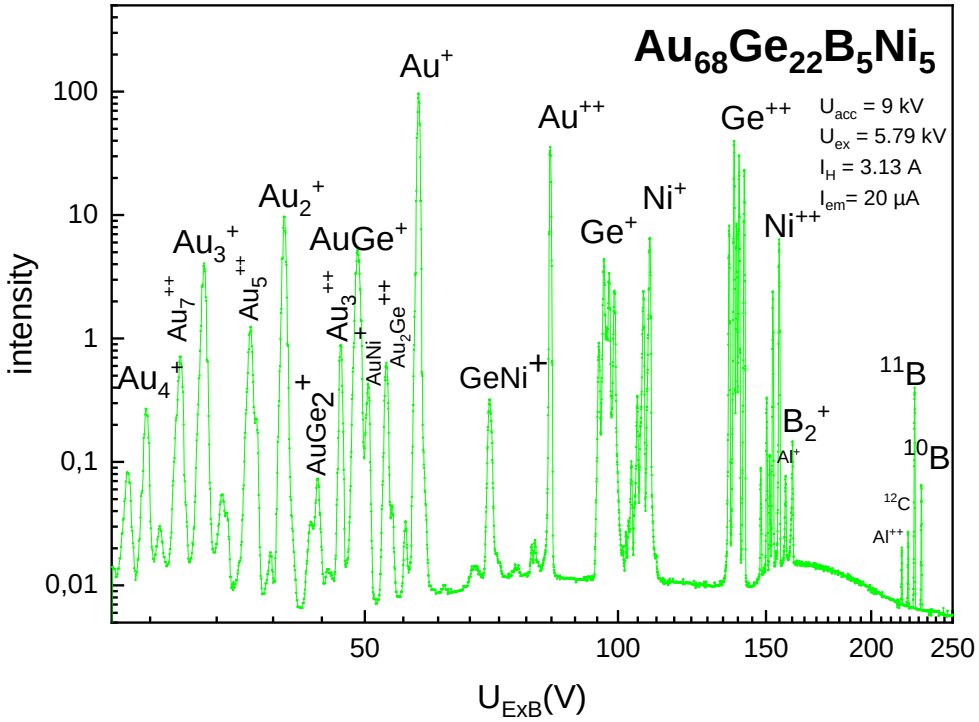


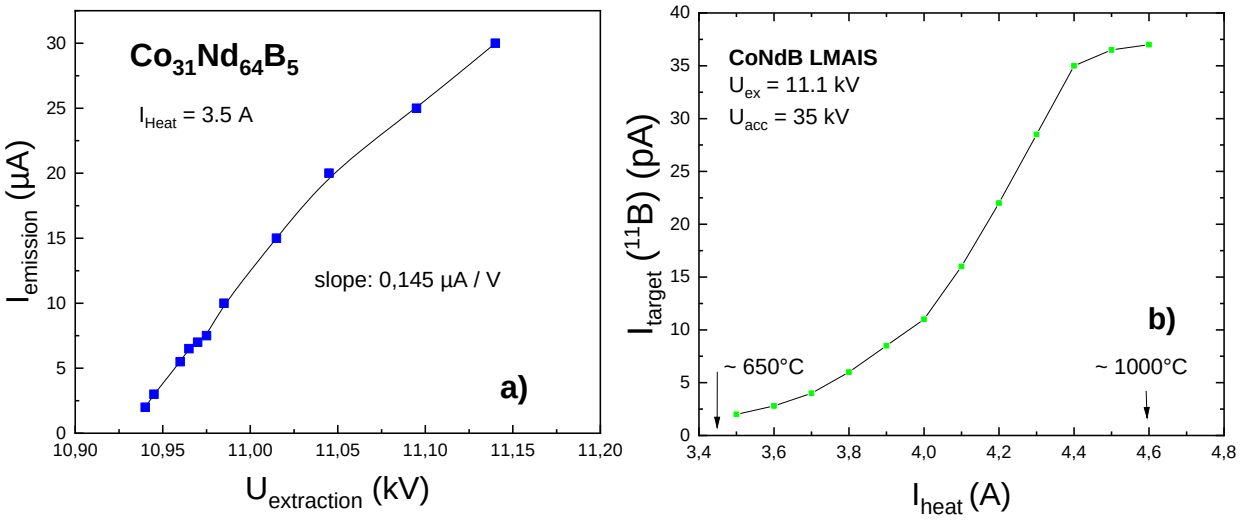

Figure 2: Mass spectrum of an $Au_{68}Ge_{22}B_5\,Ni_5$ LMAIS.

The performance of the source is limited to a small temperature window. At lower heating the boron emission is strongly decreasing and at too high temperatures source material will be evaporated. Also the source needs a higher emission current (20 µA) for a stable operation - unfavorable due to chromatic aberrations. The total life time of the source was limited to about 10 hours whereat a restarting after an interruption becomes more and more complicated.

## *C. $Co_{31}Nd_{64}B_5$*

The most suitable source properties among the investigated candidates were found with the emitter wetted with a $Co_{31}Nd_{64}B_5$ alloy and prepared on a tungsten tip. The initial point for this source development was the previous evolved very stable and reliable working $Co_{36}Nd_{64}$ LMAIS ($T_{melt}$ = 566°C) [11]. Also this source needs some temperature restrictions to not exceed 700°C due to the formation of stable NdB which remains solid also above 1000°C as well as out diffusion and evaporation of boron. On the other side a minimum temperature of 600°C should be exceeded to get the alloy liquid. The emission current should be limited to 30 µA to avoid additional heating of the tip by secondary electron bombardment and also by the emission process itself. The I-V characteristics of the

$Co_{31}Nd_{64}B_5$ LMAIS obtained in the VELION system is shown in Fig. 3a. The slope of 0.145 µA/V is steeper than known from other sources. The reason is probably the large distance between tip and extractor of 3 mm resulting in high extraction voltages. A comparable value of 0.1 µA/V was also reported for a $Pd_{70}As_{16}B_{14}$ source at extraction potentials between 8.6 and 9 kV [12] which is explained with the attendance of arsenic in the source material. The target current of $^{11}B$ as a function of the heating current is presented in Fig. 3b. The emission starts at about 650° C and at approximately 1000° C the emission comes into a saturation caused mainly by alloy evaporation. A working temperature higher than 700°C leads to a strong reduction of the source life time.



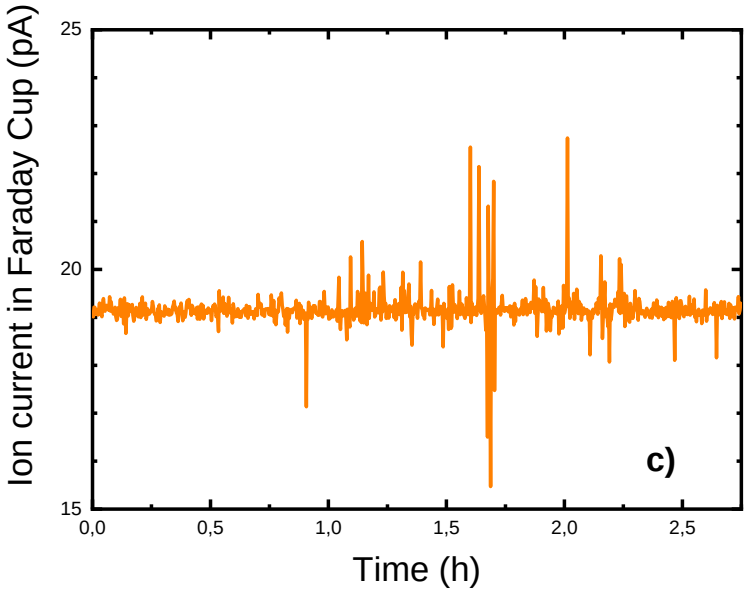



Figure 3: a) I-V characteristics of the $Co_{31}Nd_{64}B_5$ LMAIS in the VELION system, b) current of $^{11}B$ as a function of heating current, c) $^{11}B$ target current stability of (19.2 ± 0.4) pA (with spikes ± 3.7 pA) over 2.5 hours ($\Delta t_{meas}$ = 10 s).

The target current stability of the CoNdB LMAIS for the $^{11}B$ beam was measured every 10 s over a time of 2.5 hours. It was determined to (19.2 ± 0.4) pA apart from some spikes with a maximum deviation of ± 3.7 pA, see Fig. 3c. The source could be operated in a stable mode over a duration of more than 600 µAh at an emission current of about 2 µA giving a total life time of more than 300 h.

The mass spectrum of the ion source was measured in our test-FIB system [7] and is presented in Fig. 4. The applied operation parameters are plotted in the inset. The $^{11}B$ intensity reaches as more than 5% of $Nd^{++}$ and 10% of $Co^{+}$ or $Co^{++}$. Due to contamination of the starting materials, a few additional lines in the percentage intensity range or less can be seen in high-resolution mass spectroscopy. Applying this source in the VELION system also the doubly charged boron species could be found shown in Fig. 5. The current in this system is measured in a Faraday cup, i.e. the current for double charged ions must be divided by two times the elementary charge to get the number of particles per second. The maximum total ion current without mass separation was 4 nA by 10 µA emission current in the VELION system. In the case of the light ion boron the ionization energy from single to double charge state increases only from 8.3 eV to 25.1 eV [13], whereas in previous experiments for a $Ga_{35}Bi_{60}Li_5$ LMAIS [14] this increase for lithium was from 5.4 eV to 75.6 eV [13] whereby doubly charged Li ions could not be detected.

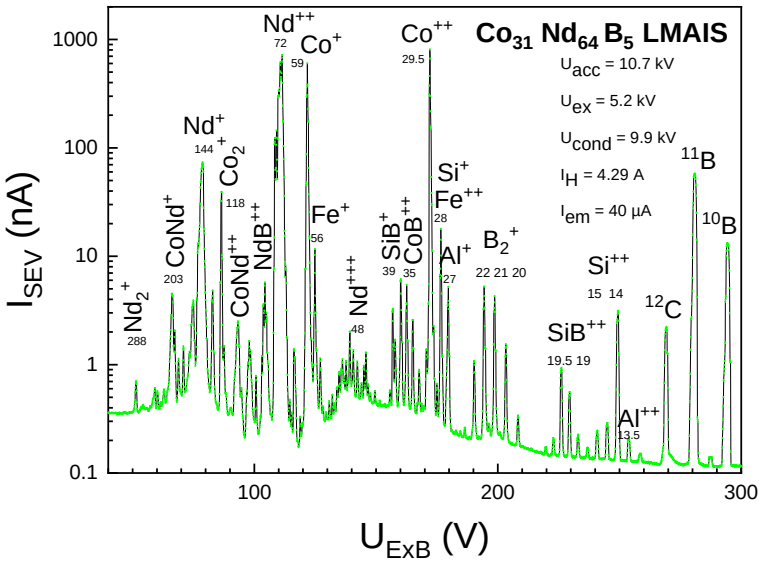



Figure 4: Mass spectrum of a $Co_{31}Nd_{64}B_5$ LMAIS. The current $I_{SEV}$ in the test-FIB was measured with a secondary electron multiplier.

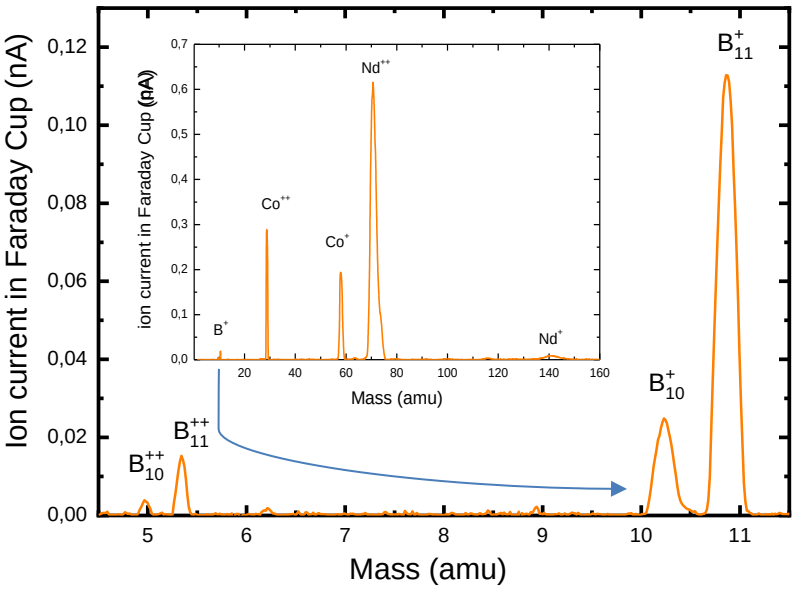
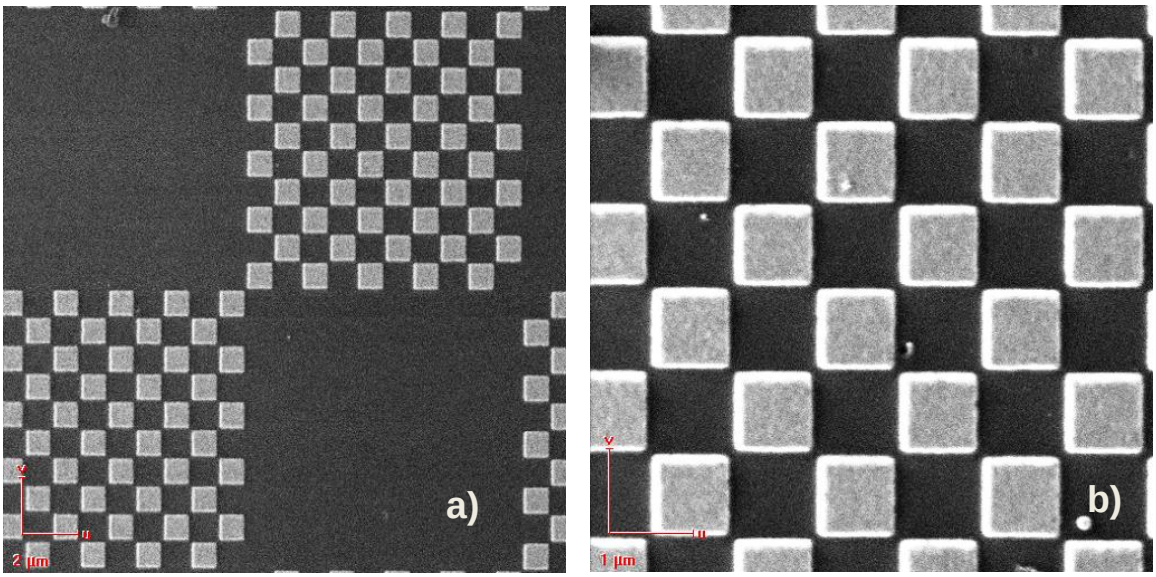



Figure 5: Mass spectrum of a $Co_{31}Nd_{64}B_5$ LMAIS obtained in the VELION system. The main part is shown in the inset as well as the evidence of the single and double charged well separated boron species.

Finally the FIB resolution for the $^{11}B$ beam was estimated by imaging a Chessy-test structure (Plano) depicted in Fig. 6. For an emission current of 10 µA at an energy of 35 keV in the VELION system, the edge resolution was evaluated to (30 ± 5) nm for an $^{11}B$ FIB with 5pA ion current, see Fig. 6a. The application of a $Co_{31}Nd_{64}B_5$ LMAIS gives also the opportunity to use also heavier elements in the FIB beside boron. The application of a $Co_{31}Nd_{64}B_5$ LMAIS allows also the opportunity to use heavier projectiles like Co or



Nd. As an example in Fig. 6b the imaging of a Chessy-test structure using a $^{142}Nd^{++}$ FIB with 44 pA is presented.

Fig. 6. a) Imaging of a Chessy-test structure with an $^{11}B$ FIB with 5 pA. b) Imaging of a Chessy-test structure with 44 pA of $^{142}Nd^{++}$.

# III. SUMMARY AND CONCLUSIONS

For the primary aim of a local p-type doping in the nano-scale boron containing alloys were tested in order to fabricate a long lasting and stable B containing LMAIS. Some possible candidates namely $Co_{31}Nd_{64}B_5$, $Au_{77}Si_{18}B_5$ and $Au_{70}Ge_{25}B_5$ and $Au_{68}Ge_{22}Ni_5B_5$ were investigated. The first one exhibited the best performance and was additional tested under real practical conditions in a VELION FIB-SEM system (Raith GmbH). The source life time was longer than 600 µAh and a first characterization showed a lateral resolution of (30 ± 5) nm so far. This LMAIS is suited for several mass-filtered FIB applications like implantation, high rate sputtering, surface patterning or ion lithography. The fast switching between the certain ion species in the case of a $Co_{31}Nd_{64}B_5$ LMAIS from B – very light, suitable for ion lithography [15] or writing p-type doping [3] to Co – medium mass for applications in the field of nano-magnetics or $CoSi_2$ ion beam synthesis of conductive nano-structures on Si [16] and finally to heavy Nd double charged ions for ion sputtering. The latter one reveals to be problematic to separate all isotopes leading to a reduced lateral resolution. The change between ion species can be done in seconds and leads to remarkable expansion of the application spectrum of FIB technology.

## ACKNOWLEDGMENTS

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