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## AN MeV FACILITY FOR MATERIALS RESEARCH

A. POLMAN, A.M. VREDENBERG, W.H. URBANUS, P.J. VAN DEENEN, H. ALBERDA, H. KROP, I. ATTEMA, E. DE HAAS, H. KERSTEN, S. DOORN, J. DERKS, J. TER BEEK, S. ROORDA, R. SCHREUTELKAMP, J.G. BANNENBERG and F.W. SARIS

*FOM-Institute for Atomic and Molecular Physics, Kruislaan 407, 1098 SJ Amsterdam, The Netherlands*

A facility consisting of two Van de Graaff-type MV accelerators for materials processing and characterization has been developed in collaboration with High Voltage Engineering Europa B.V. A newly developed heavy-ion implanter is used for generation of several-hundred- $\mu$ A mass-analysed ion beams in the energy range from 10 keV up to 2 MeV. Beams of a wide range of elements can be extracted from three different types of ion sources, which can be exchanged in minutes using a high-pressure load-lock system. Other important new features include: integrated gas system, specially developed ion optics and injection system, mass analysis at high voltage level and an X-ray intensity level  $< 0.1$  mrem/h. This accelerator is connected to two beam lines for (1) ion mixing, including in situ sample analysis using RHEED, RBS, channeling and NRA, and (2) ion implantation of up to  $10 \times 10$  cm<sup>2</sup> samples in a class 100 clean room environment.

In addition, the facility comprises a 2 MV system dedicated to sample analysis using RBS, channeling, NRA and PIXE. By the use of a magnet configuration in the acceleration tube also for this 2 MV accelerator an X-ray intensity level  $< 0.1$  mrem/h is obtained. The complete facility enables one to perform a wide variety of experiments concerning materials modification and analysis with ion beams in the first  $\mu$ m of semiconductors, metals and other materials.

### 1. Introduction

High-energy (MeV) ion implantation in metals and semiconductors has recently become a new and interesting topic in materials research [1-6]. Compared to the now well-established medium-energy (up to a few hundred keV) implantation technique, deeper and thicker surface regions can be modified using high energy ion beams, resulting in novel and unique structures with new possibilities and technological important applications. Now that high-current, high-energy accelerators have become available these new features can readily be explored.

For example, nitrogen, carbon or titanium implantation in metals can be employed to improve macroscopic surface properties such as hardness, friction, wear- and corrosion resistance [7,8]. Changes in these properties become more pronounced and can be determined more accurately when thicker layers are modified. Also, MeV implantation can be employed for ion mixing of multi-layer structures to form micron thick metastable alloy films.

Moreover, a huge range of applications of MeV ion beams seems to lie in semiconductor technology [1,2]. Through this technique it is possible to produce buried layer structures several microns beneath the surface having materials characteristics different from those of the surface. Buried doped layers [3,4], as well as buried

insulating [5] or conducting layers [6] can be fabricated and consequently a whole range of novel device structures can be developed.

Apart from ion implantation in metals and semiconductors new applications can be found in ceramic and superconductivity technology. Using ion implantation in superconducting films, the electrical properties can be modified locally [9]. Also, high-dose, high-energy oxygen ion mixing could be employed for formation of thick high- $T_c$  ceramic oxide films.

Also from a fundamental point of view MeV implantation offers interesting possibilities. Now that thicker layers can be modified, macroscopic analysis techniques can be employed for a thorough study of materials properties of a wide variety of samples. Furthermore, MeV ion implantation offers the possibility to distinguish between nuclear and electronic effects on processes such as ion beam assisted solid-phase epitaxial growth [10], damage formation [4] and adhesion of thin films on substrates [11].

MeV accelerators have been constructed commercially in various concepts during the last two decades and have mainly been used for nuclear reaction and analysis purpose. The use of MeV beams for ion implantation implies severe demands for the accelerator's technical specifications:

- (1) A wide variety of ion species, such as P, As, B, noble gases, O, N, Si as well as metal species,

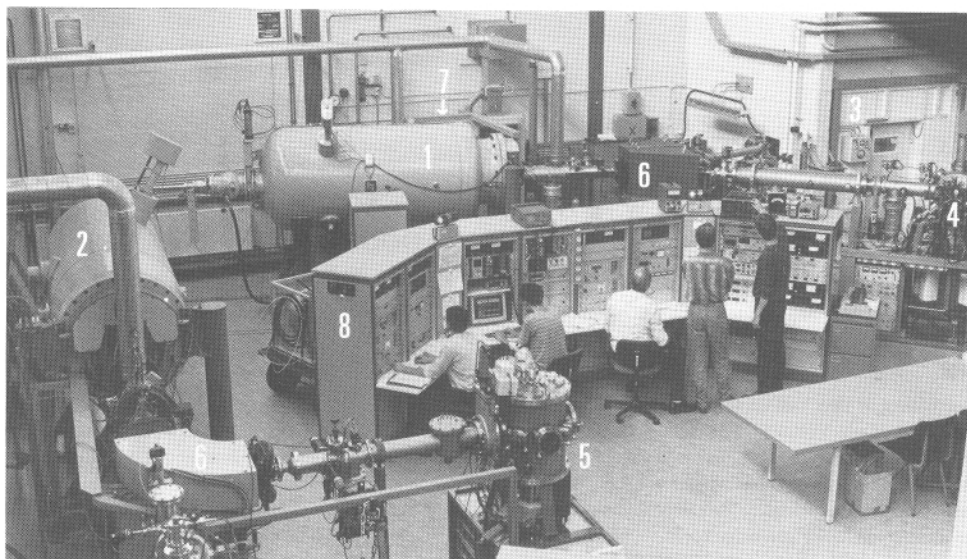


Fig. 1. Photograph of the accelerator hall. 1) 1 MV accelerator, 2) 2 MV accelerator, 3) clean room with large-area implantation chamber, 4) UHV system for ion mixing and ion implantation, 5) RBS analysis chamber, 6) analysing magnets, 7) gascabinet, 8) integrated control cabinet for 1 and 2 MV machines.

should be available. Hence, the ion injection system needs to be compatible with various types of ion sources.

- (2) For high implantation efficiency, source exchange and servicing needs to be possible quickly. This requires special facilities for Van de Graaff-type machines where the source is situated in a pressure vessel.
- (3) Ion currents of several 100  $\mu\text{A}$  should be available. This implies efficient mass analysis of species from the ion source prior to acceleration, to enhance the acceleration efficiency.
- (4) For efficient use of expensive laboratory floor space, the X-ray background intensity needs to be reduced to such a level that no concrete shielding is necessary.

In this paper an MeV facility is described in which all these requirements are effectively met. It consists of two single-ended Van de Graaff-type accelerators: a high-current 1 MV heavy-ion accelerator used for ion implantation in metals and semiconductors and a 2 MV accelerator operated for sample analysis using Rutherford backscattering spectrometry (RBS), channeling, nuclear reaction analysis (NRA) and particle induced X-ray emission (PIXE) [12].

The facility has been designed in close collaboration with High Voltage Engineering Europa (HVEE). It is based on Van de Graaff technology, but in addition several new features were developed. The collaboration started in 1985 and has resulted in a series of publications on various specific elements under construction

[13–16]. By the end of 1987 the facility was completed and the first test sequences were finished successfully. It is now employed for materials research at the FOM-Institute. In this paper we give an overview of the total equipment, the new features and the experiences during operation. Fig. 1 gives an overview of the complete facility, situated on an area not larger than  $12 \times 14 \text{ m}^2$ .

## 2. The 1 MeV ion implantation facility

The accelerator is a single-ended Van de Graaff-type machine with a high voltage terminal in a pressure vessel containing an insulating gas mixture of 20%  $\text{SF}_6$  and 80%  $\text{N}_2$  at a total pressure of 6 bar. The total current available at 1 MV terminal level for acceleration is 700  $\mu\text{A}$  and the total electrical power available at the terminal for various power supplies is 3 kW. Hereafter the new features will be described.

### 2.1. Ion sources, source exchange system and injector

The 1 MV implanter can be operated with three different types of ion sources. A cold cathode Penning source [17] can be used for most nonreactive gases. Because of its high discharge voltage and low operating pressure it is well suited for the production of multiply charged ions. Ions from chemically reactive gases can be obtained from a newly developed microwave ion source [14–16]. Since the source contains no hot filament, it can be operated for several hundred hours, during which

stable, high current beams are easily generated. A sputter ion source [17] is available for generation of species from solid targets, especially metals.

In order to allow a quick exchange of different source types, a unique ion source exchange system has been developed [13]. It is positioned at the rear of the high pressure vessel and enables source exchange within minutes, without breaking the high pressure in the main vessel. All ion sources have a dedicated extractor configuration and are fixed to a standard flange which fits to an accepting flange on the injector end. This flange contains all electrical connections, the gas feedthrough and vacuum gaskets. In a source loading chamber, an ion source can be mounted onto a rod whereafter it can be transferred to its position on the injector system. Via the main gas handling system which is described in section 2.2 the loading chamber is connected to both high pressure and vacuum systems.

In the high voltage terminal the source deck is permanently equipped with power supplies for all three different types of ion sources. Because of the varying and extreme conditions of high pressure, vacuum, sparking, limited space and limited power available, special attention has been paid to the development of these supplies. They are well stabilized against external variations within one tenth of a percent, suitable for telemetry and telecontrol from earth potential and extremely well protected against any conceivable form of overload during breakdown or arcing of the accelerator.

Fig. 2 is an overview of the source deck showing the compact configuration of power supplies for microwave-, Penning- and sputter sources as well as the ion source gas cylinders. As compactness is the dominant requirement, all supplies make use of the so-called switched-mode technique. The available power of 115 V-400 Hz is first rectified into a common 300 V dc supply. In every supply this dc voltage is converted into a 50 kHz square wave. Output voltage or current regulation is performed by switching is performed by switching the on/off ratio of the square wave. As switching is done by nearly ideal MOSFET switches, the power dissipation is very low compared to that of the traditional dissipative series regulation systems. This enables compact construction without undue heat problems. Although the requirements for output voltage and current are different, all supplies are based on the same design and technology and most of the control and switching circuitry is identical, which facilitates reproduction and maintenance at low cost.

To obtain large ion currents it is necessary to do mass analysis of species extracted from the ion source at the terminal level, before acceleration. This enables one to make use of the maximum acceleration current capacity for the desired ion species. This (coarse) separation is done by using a Wien filter with a mass-separation capability of  $m/\Delta m \approx 15$ . Details are given in earlier publications in which the complete ion-optical design of the injector system is described [13,14].

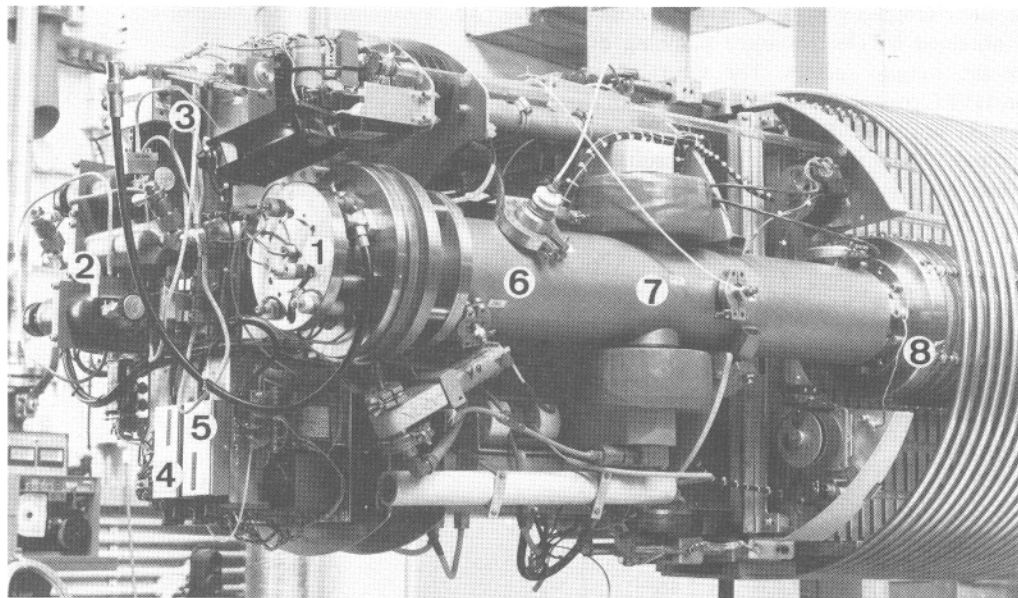


Fig. 2. Overview of source deck and injector system: (1) plug-in ion source, (2) gas cylinders, (3,4 and 5) power supplies for microwave-, Penning-, and sputter ion sources, (6) einzel lens, (7) Wien filter, (8) acceleration tube.

## 2.2. Integrated source- and insulating-gas system

Another special feature of this implanter is the integrated system for handling source gas and insulating gas, used for source exchange and gas supply. Fig. 3 shows a schematic overview of this system. Source gas is supplied from a gas manifold in a gas cabinet on earth potential which contains purge facilities and satisfies safety requirements for toxic gases. Gas supply into the terminal from earth potential offers the possibility to subsequently use a large number of different gases without opening the pressure vessel. From the cabinet small and controllable quantities of gas can be fed into a reservoir near the ion source. In addition to this multi-use reservoir, two permanently filled high-pressure gas cylinders are placed near the ion source (see fig. 2). Using a gas-selection switch controlled from earth potential, the proper gases can be supplied to the ion source. To bridge the voltage difference in the gas tube between source and baseplate an insulating  $\text{Al}_2\text{O}_3$  tube was chosen. During operation of the accelerator this tube is filled with insulating gas at the tank pressure. Therefore the source gas system is integrated with the insulating gas system. A complete gas exchange cycle takes about 30 min for nontoxic gases and somewhat longer when tubes have to be purged. Also, ion sources can be easily exchanged with the aid of this gas system.

## 2.3. Beam lines

Two beam lines are connected to the 1 MV heavy-ion implanter. The first comprises a UHV system from HVEE for ion implantation and ion mixing studies of small ( $\approx 1 \text{ cm}^2$ ) samples [18]. Implantation dose uniformity is obtained by electrostatic scanning of the beam over the sample surface. The target chamber contains an HVEE precision goniometer [19] with three axes of rotation and three degrees of translational freedom. It is compatible with a sample holder suitable for sample exchange using a high-vacuum load-lock system.

An electron gun evaporation unit for multilayer thin film deposition is incorporated. In situ sample analysis techniques include reflective high energy electron diffraction (RHEED), RBS, channeling and NRA, whereas an X-ray detector for PIXE may be added.

The second beam line is connected to a large-area implantation facility situated in a class 100 clean room. Using mechanical scanning, batches of up to seven  $10 \times 10 \text{ cm}^2$  samples can be implanted. Fast feedback between current measured on target and scan speed enables good uniformities over the large area wafers. The acceleration voltage range over which the accelerator can be operated to yield stable, high-current ion beams is between 100 kV and more than 1000 kV. To extend this range to lower voltages, a decelerating lens system was developed for the large-area implantation chamber. Using a high voltage (up to 100 kV) power supply, ions can be decelerated to energies as low as 10 keV before they reach the target.

## 3. The 2 MV facility for RBS, channeling and NRA

To enable high-throughput RBS analysis, a second accelerator was installed. It comprises a standard single-ended 2 MV Van de Graaff-type accelerator with a pressure vessel containing an insulating gas mixture of 20%  $\text{SF}_6$  and 80%  $\text{N}_2$  at a total pressure of 10 bar. The high voltage terminal comprises simple power supplies and an rf-type ion source with two high-pressure source-gas cylinders containing He and H. The new features of this facility are described hereafter.

### 3.1. X-ray intensity suppression

One of the main hazards of a standard Van de Graaff accelerator is its high level of X-ray radiation. X-rays are produced when electrons, generated from ion-gas or ion-electrode collisions and accelerated towards higher voltage regions, hit solid material to gener-

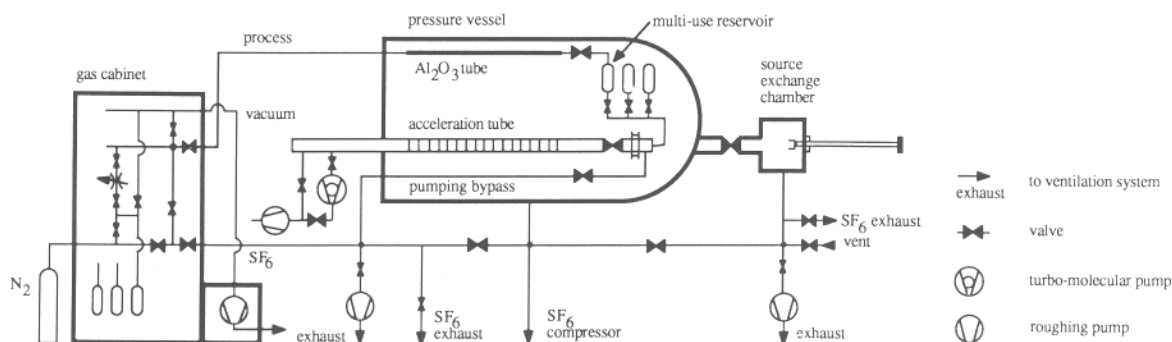


Fig. 3. Schematic overview of the integrated system for source gas and insulating gas, used for source exchange and gas supply.

ate bremsstrahlung. This problem is solved in both the 1 MV and 2 MV accelerators by positioning a series of magnets around the central holes in the electrodes in the acceleration tube. The integral magnetic field which is thus created bends the path of the electrons, such that they are stopped only a few electrodes away from where they originated. Computer simulation of the electron trajectories was employed to design a magnet configuration such that the maximum energy gain of the electrons is below 130 keV [20]. Continuous X-ray intensity monitors indicate that the X-ray dose rate equivalent at 10 cm outside the vessel wall never exceeds 0.1 mrem/h when the machines are operated, even when high ion currents (few hundred  $\mu\text{A}$ ) are transported.

For the 2 MV accelerator also the influence of the magnetic field on the ion paths was evaluated. Although the mass difference between electrons and ions is very large, the magnetic forces have a nonnegligible effect on low-mass ion trajectories. This disturbance is of importance as for easy operation of the accelerator the ion beam should enter the beam line near its center and parallel to the central axis. In addition, during traversal of the acceleration tube, the maximum deviation from the central axis should be small compared to the radius of the apertures in the electrodes, to prevent ions from hitting the electrodes. In order to fulfill these requirements the spatial dependence of the direction of the magnetic field in the acceleration tube was optimized. Ion trajectories were calculated using computer simulation and optimization.

In the optimum configuration the acceleration tube is divided into regions in which the direction of the magnetic field is rotating either clockwise or counterclockwise around the tube axis. Fig. 4 shows the corresponding calculated beam excursion in the tube for a  $\text{H}^+$  beam accelerated to 200 keV. Because all ion trajec-

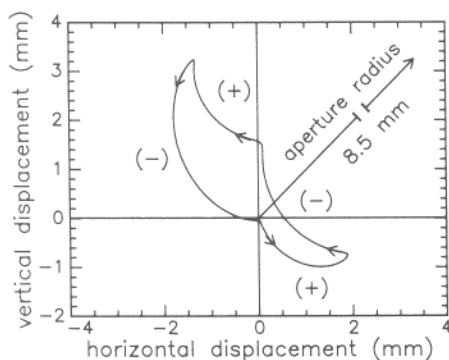


Fig. 4. Ion trajectory calculation for a  $\text{H}^+$  beam accelerated to 200 keV in a tube containing the optimized magnet configuration. The beam deviation from the central axis is represented in the plane normal to the axis. (+) and (-) denote the clockwise and counterclockwise rotation of the magnetic field in the tube, respectively.

tory deviations scale with a factor  $(ME)^{-1/2}$ , with  $M$  and  $E$  the mass and energy of the ion respectively, a calculation for a 200 keV  $\text{H}^+$  ( $M = 1$ ) beam is a worst case scenario. The beam enters and leaves the tube on axis, with the exit angle near zero: 0.4 mrad. (The latter conclusion cannot be drawn from fig. 4, but follows from the total ion trajectory calculation [20].) As can be seen, the maximum off-axis beam deviation is only 3.5 mm, whereas the electrode aperture radius is 8.5 mm, preventing the ion beam (with maximum beam envelope radius 2.5 mm [20]) from hitting electrode material.

### 3.2. Ion energy stabilization and operation simplicity

The 2 MV accelerator and the connected RBS facility are characterized by a high degree of simplicity both in use and setup. Ion energy stabilization and ion beam manipulation are controlled with great ease from a simple control cabinet. For both 1 MV and 2 MV accelerators a high voltage stabilizer was developed, such that operation essentially requires control of only two knobs. Actual stabilization can be chosen from three different modes.

(1) In manual operation, the terminal voltage is set by the charge which is sprayed onto the belt. The voltage is determined by a balance between the setting of the belt charge power supply and the current drawn from the terminal by the column resistance, the emitted ions and a corona drain circuit. The corona current drain is kept within its electronic controllable range of operation by an automated moving of the corona rod.

(2) As soon as enough corona current is flowing, the voltage can be stabilized by choosing the generating voltmeter (GVM) mode. Stabilization is achieved by feeding back the signal from the GVM and a capacitive pickup device. Low-frequency voltage variations (up to a few Hz) are counteracted by controlling the charge rate of the belt, whereas correction for higher-frequency variations (up to a few hundred Hz) is obtained by controlling the corona current by a triode in the corona drain circuit.

(3) An even higher degree of energy stabilization is achieved by operating the accelerator in the slit stabilization mode. Once the ion beam is hitting the energy defining slit behind the analysing magnet, the system searches for that particle energy which yields an equal ion current on both sides of the slit. The acceleration voltage is then determined by the magnetic field and automatically keeps track when the magnetic field is varied.

The ion optics for this accelerator system are kept very simple as well. The ion beam is focussed by the acceleration tube at a position in front of the analysing magnet, which in its turn images the focal point near the target. The beam focus can be controlled by simple adjustment of the extraction voltage. As described in



the preceding section the electron suppressing magnetic field is designed such that the ions leave the tube near and parallel to the central axis. Two simple sets of deflection plates suffice for fine tuning of the beam position.

### 3.3. Scattering chamber

The 2 MV accelerator is connected to a HVEE scattering chamber for RBS, channeling, NRA and PIXE. After energy selection in the analysing magnet the ion beam passes through two diaphragms for precise angle selection. He<sup>+</sup> currents on target of over 100 nA with an angular spread of less than 0.1° can be obtained. The scattering chamber contains a four-degree-of-freedom manipulator and goniometer compatible with a 50 mm diameter sample holder. Backscattering analysis is performed using a standard Si surface barrier detector, which can be cooled to -20°C to obtain an energy resolution as low as 12 keV.

The authors would like to acknowledge all the collaborators concerned in this project. This work is part of the research program of the Stichting voor Fundamenteel Onderzoek der Materie (FOM, Foundation for the Fundamental Research on Matter) and was made possible by financial support from the Nederlandse Organisatie voor Wetenschappelijk Onderzoek (NWO, Netherlands Organisation for the Advancement of Pure Research) and the Stichting Technische Wetenschappen (STW, Netherlands Technology Foundation).

### References

- [1] D.C. Ingram, Nucl. Instr. and Meth. B12 (1985) 161.
- [2] D. Pramanik and A.N. Saxena, Nucl. Instr. and Meth. B10/11 (1985) 493.
- [3] P.F. Byrne, N.W. Cheung, S. Tan, C. Hu, Y.C. Shih, J. Washburn and M. Strathman, Mater. Res. Soc. Proc. 27 (1984) 253.
- [4] M. Tamura, N. Natsuaki, Y. Wada and E. Mitani, Nucl. Instr. and Meth. B21 (1987) 438.
- [5] R.F. Pinizzotto, Mater. Res. Soc. Proc. 27 (1984) 265.
- [6] A.E. White, K.T. Short, R.C. Dynes, J.P. Garino and J.M. Gibson, Appl. Phys. Lett. 50 (1987) 95.
- [7] G.K. Hubler and F.A. Schmidt, Nucl. Instr. and Meth. B7/8 (1985) 151.
- [8] G. Dearnaley, Nucl. Instr. and Meth. B7/8 (1985) 158.
- [9] G.J. Clark, A.D. Marwick, R.H. Koch and R.B. Laibowitz, Appl. Phys. Lett. 51 (1987) 139.
- [10] R.G. Elliman, J.S. Williams, W.L. Brown, A. Leiberich, D.M. Maker and R.V. Knoell, Nucl. Instr. and Meth. B19/20 (1987) 435.
- [11] J.E. Griffith, Y. Qiu and T.A. Tombrello, Nucl. Instr. and Meth. 198 (1982) 607.
- [12] W.K. Chu, J.W. Mayer and M.A. Nicolet, Backscattering Spectrometry (Academic Press, New York, 1978).
- [13] R. Koudijs, P. Dubbelman, W.H. Urbanus and J.G. Bannenberg, Nucl. Instr. and Meth. B21 (1987) 296.
- [14] W.H. Urbanus, J.G. Bannenberg, S. Doorn, S. Douma, J. Ishikawa, F.W. Saris, R. Koudijs and P. Dubbelman, Nucl. Instr. and Meth. A267 (1988) 237.
- [15] J. Ishikawa, Y. Takeiri and T. Takagi, Rev. Sci. Instr. 55 (1984) 449.
- [16] J. Ishikawa, S. Douma, W.H. Urbanus, S. Doorn, J.G. Bannenberg and F.W. Saris, Proc. 10th Symp. on Ion Sources and Ion Assisted Technology, ed. T. Takagi (Kyoto University, Kyoto, 1986) p. 105.
- [17] H. Baumann and K. Bethge, Nucl. Instr. and Meth. 189 (1981) 107.
- [18] J.F.M. Westendorp, P.K. Rol, S. Doorn, H. Kersten, J. ter Beek, J. Derks, F.W. Saris, W.J. van Kilsdonk and R. Koudijs, Nucl. Instr. and Meth. B17 (1986) 66.
- [19] R.M. Tromp, H.H. Kersten, E. Granneman, F.W. Saris, R. Koudijs and W.J. van Kilsdonk, Nucl. Instr. and Meth. B4 (1984) 155.
- [20] W.H. Urbanus, J.G. Bannenberg, F.W. Saris, R. Koudijs and P. Dubbelman, to be published.