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## Accelerator SIMS at PSI/ETH Zurich

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### Abstract

The accelerator SIMS system at PSI/ETH Zurich has been completed and has been run successfully in first tests. A new ion source has been added to the existing AMS facility. The main concept of the ion source is the suppression of the contamination of the sample and of the secondary ion beam. All parts close to the sample have therefore been gold plated. With a commercial mass filtered Cs gun as used for secondary ion mass spectrometry (SIMS) a pure Cs ion beam is obtained for sputtering. In first experiments some routinely used AMS techniques have been shown to be very effective for accelerator SIMS as well. In many cases a fast beam-pulsing system allows normalization of trace element yields to the substrate current. In order to avoid contamination of the accelerator SIMS chamber, a pilot beam from the adjacent AMS sputtering source can be used for tuning of the accelerator mass spectrometer to the trace element of interest. Since the AMS ion source yields a more intense current, tuning becomes easier and, in addition, a good reference spectrum for the gas ionization counter is obtained. First results are presented.

### 1. Introduction

Accelerator secondary ion mass spectrometry (accelerator SIMS) is a very promising technique for the detection of trace elements in ultrapure materials [1]. Compared with ordinary SIMS it has two major advantages: (i) molecules are broken up in the stripper of the tandem accelerator leading to a pure atomic mass spectrum; (ii) thanks to their high energy, particles can unambiguously be identified with virtually no background.

Accelerator SIMS tests performed in Zurich in 1992 [2] using an AMS sputtering chamber produced very promising results concerning the detection of stable trace elements in pure materials. However, it was also shown that the major problem of this technique is contamination of the sample in the sputtering chamber. This effect especially affects the detection limits of elements present in stainless steel (Fe, Cr, Ni, Mo). A new UHV ion source with special features designed to prevent contamination has therefore been built and added to the injection beam line of the accelerator. First experiments have shown that the system as a whole works well and that the measurement techniques of AMS are also well suited for accelerator SIMS.

### 2. Setup

The part of the AMS facility at PSI/ETH Zurich relevant to accelerator SIMS is shown in Fig. 1. The new ion source has been connected to the existing heavy ion injector by an additional small electrostatic deflector.

The main concept of the new accelerator SIMS chamber is the suppression of sample contamination during the sputtering process. Fig. 2 shows the chamber with the Cs ion gun and the target region. The chamber itself is at ground potential, while the target is at high voltage (–30 kV). The target holder is mounted on an  $x$ – $y$  stage with an insulation capable of 40 kV potential difference. The  $x$ – $y$  stage is driven by two in-vacuum stepping-motors. To avoid sample cross contamination a target cassette containing only one sample is introduced into the sputtering chamber via a load lock system. In order to keep contamination of the extracted ion beam as low as possible, the entire environment of the target is plated with a 20  $\mu\text{m}$  thick layer of pure gold (99.99%). This should reduce the background level of any element but gold. Gold has been chosen because of the simplicity of the plating process. If contaminated, all parts can be cleaned and replated with ease. For more details see Ref. [3].

The Cs sputter gun is a commercial SIMS product from ATOMIKA [4]. Like the target, the gun and its electronic control system are at –30 kV. The Cs gun controls can be operated mechanically with plexiglass rods. Computer con-

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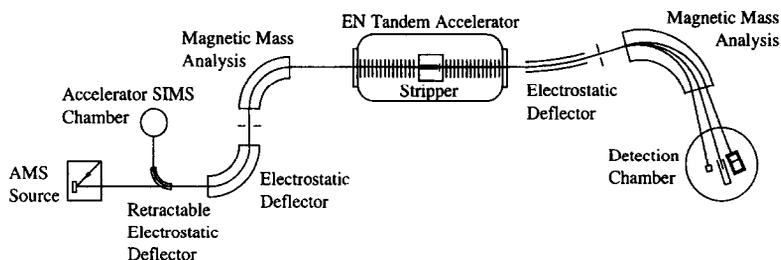


Fig. 1. Part of the AMS facility at PSI/ETH Zurich relevant to accelerator SIMS experiments.

control of certain parameters (e.g., beam on/off,  $xy$ -deflection, gated field) is also possible via optical links. The gun is equipped with an  $E \times B$  filter and a  $1^\circ$  beam bend for neutral particle suppression leading to a pure Cs ion beam. The sputter energy is variable between 15 and 3 keV. With different apertures it is possible to vary the beam spot size and the corresponding current. These parameters can thus be optimized for different applications such as bulk analysis, imaging and depth profiling.

For the evaluation of the spot size the Cs beam has been raster scanned over a target by electrostatic deflection of the Cs beam. The target consisted of a Au grid clamped to a Si wafer. For a given Cs beam intensity, the net current measured on the target is different for Si and Au due to the different secondary electron emission. The current on the target has been measured and plotted against beam position (Fig. 3). With this method we have shown that at an energy of 10 keV a 400 nA Cs beam can be focused to a spot of  $150 \mu\text{m}$  diameter. It is also possible to obtain 5 nA focused to  $10 \mu\text{m}$  diameter. With the smallest aperture the beam can be focused down to  $2 \mu\text{m}$ . The gun is thus well suited for imaging.

If sputtering speed is constant, a depth profile of a trace element can be obtained by drawing the measured trace element yield versus sputtering time. For good depth resolution, however, it is essential to produce a flat sputter crater with a low sputtering energy. Therefore, it is possible to reduce the Cs beam energy down to 3 keV. In

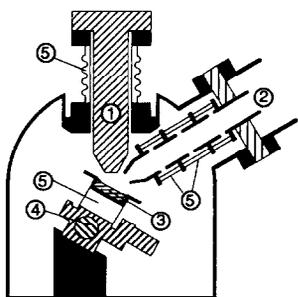


Fig. 2. Sputtering chamber for accelerator SIMS experiments: (1) Cs ion gun, (2) secondary ion extraction, (3) target, (4)  $x$ - $y$  table, (5) insulators.

addition, the raster scan control produces a gate signal for exclusion of the outer part of the scan field.

The secondary ion extraction system consists of an immersion lens with four electrodes that accelerates the negative ions from the target to 30 keV. An  $x$ - $y$  steerer followed by a quadrupole doublet and another  $x$ - $y$  steerer focuses the beam to the object point of a small spherical electrostatic deflector (beam radius 12 cm). The image point of this deflector corresponds to the object point of the heavy ion injector consisting of a  $90^\circ$  spherical electrostatic deflector and a  $90^\circ$  dipole magnet. With slit apertures of 3 mm the injector has a mass resolution of  $m/\Delta m = 370$ . The achievable magnetic field allows the injection of any stable isotope into the accelerator [5].

The accelerator terminal voltage is set to 5 MV. At the high energy end of the accelerator the beam passes through a  $15^\circ$  electrostatic deflector with a radius of 5.8 m. The electrostatic analysis is set to a value of  $E/q$  correspond-

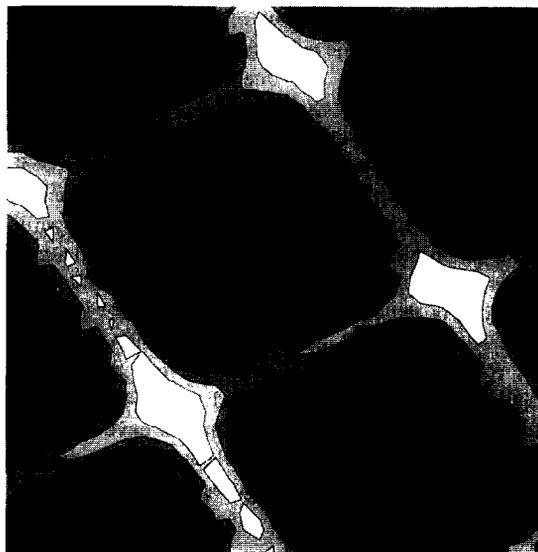


Fig. 3.  $180 \mu\text{m} \times 180 \mu\text{m}$  image of a 300 mesh Au grid clamped to a Si wafer. The net current measured on the target is plotted against Cs beam position. The Cs current of 5 nA is focused to a diameter of  $10 \mu\text{m}$ .

ing to monoatomic ions with an energy of 40 MeV in charge state 7. A 90° dipole magnet then deflects the beam into a large detection chamber. By varying the magnetic field, the beam can be brought into different detection devices. Currents down to a few pA are measured in a Faraday cup. A gas ionization chamber can be used for unambiguous projectile identification up to a beam intensity of the order of 10 kHz. For intermediate counting rates a channelplate detector is also available. However, for the detection of trace elements in the ppm range in a Si matrix, it turns out that the Si current can be measured in the Faraday cup, whereas the trace element can be detected with the gas ionization counter. The channelplate detector is thus not used.

### 3. The pulsed detection mode

For an accurate determination of the concentration of a trace element in a Si sample both the  $^{28}\text{Si}$  current ( $\sim 700$  pA, charge state  $7+$ ) in the Faraday cup and the count rate of the trace element in the gas ionization chamber can be measured quasi-simultaneously in the detection chamber. This is realized by a fast beam-switching system, which has been used for many years to determine isotopic ratios in AMS experiments [6]. The complete system is tuned to the trace element in question. With an electrostatic pulse applied to the insulated magnet chamber at the low energy side of the accelerator,  $^{28}\text{Si}$  is injected into the tandem for 20 ms eight times per second. In the remaining time the trace element is injected. In the magnet at the high energy side of the accelerator the two elements (with different mass) have a different beam radius and can be detected in two separate detection devices. For the determination of the concentration of the trace element the probabilities for negative ion formation of the trace and the matrix element have to be taken into account. These values are known as relative sensitivity factors (RSF) from SIMS experiments and can, for example, be taken from Ref. [7]. More precise, however, is the measurement with standard samples. This reduces the determination of the concentration to a comparison of two counting rates normalized to the effective matrix element current.

Because the range of the beam path radius in the HE analysing magnet is restricted to values between 105 and 115 cm, the pulsing technique at this facility is limited to a mass difference between trace and matrix element of  $\pm 17\%$ . Thus, only the following trace elements in Si can be investigated with the pulsing technique in a straightforward way: Al, P, S.

It is possible to extend the pulsing technique to a wider mass range: trace elements around mass 56 can be investigated in the pulsed mode if  $^{28}\text{Si}_2$  is injected as the pulsed matrix beam. The  $^{28}\text{Si}_2$  molecule breaks up in the stripper of the terminal. On the high energy side of the accelerator a single  $^{28}\text{Si}$  ion in charge state  $3+$  coming from this

breakup has the same trajectory as a  $^{56}\text{Fe}$  ion in charge state  $6+$ . Therefore, trace elements with charge state  $6+$  can be detected in the gas ionization counter quasi-simultaneously with the  $^{28}\text{Si}$  ( $q = 3+$ ) current in the Faraday cup. This is possible for Ti, V, Cr, Fe, Co, Ni and Cu. However, for even charge states more care has to be taken to avoid interferences. The determination of the concentration of these transition metals in Si is of special interest in device fabrication because of their electrical activity in the semiconductor [8]. This is one of the reasons to develop a technique that can provide a sensitive measurement in this mass range.

In first experiments the phosphorus concentration in different Si samples has been determined with the technique described above. Two P doped Si samples with nominal concentrations per Si atom of  $10^{-7}$  and  $10^{-8}$ , respectively, have been analysed. With the RSF from Ref. [7] the results are within 30% of the nominal concentrations. Neither the uncertainty of the nominal concentration of the samples, nor the different ion transmissions of P and Si due to different charge state distributions after the stripper have been considered in this calculation. The lowest measured P concentration in a sample specified as undoped has been about  $6 \times 10^{-9}$ . It has to be checked if the investigated samples contain this amount of phosphorus or if this is our actual detection limit for P in Si.

### 4. Pilot beams from a second ion source

Due to the low Cs current ( $< 500$  nA) of the accelerator SIMS setup the mass analyzed secondary ion current from a Si sample is detectable in a Faraday cup only for masses 28–30 and 56–58. Therefore, a channelplate detection system will be added on the low energy side of the accelerator to allow SIMS measurements without the accelerator.

Another problem appears for the detection of rare trace elements: although the ion optical components of the beamline can be set by computer control to a specific mass and energy of the particles to be analysed, fine-tuning is still necessary for optimum transmission. This adjustment is realized by finding the highest counting rate in the gas detector. If, however, the counting rate is below 10 Hz this technique is not feasible. This problem can be overcome by the use of a pilot beam coming from the adjacent AMS ion source (Fig. 4). The beamline of the accelerator SIMS setup and that of the AMS source correspond to each other except for the first lenses and a spherical electrostatic deflector. Since this deflector is retractable, switching between the sources is very simple. This allows the use of the AMS source for tuning of the whole beamline.

In a first step, the transmission of the substrate current is optimized with the beam from the accelerator SIMS chamber. In a second step, the small electrostatic deflector is retracted and an ion beam of the same kind of substrate

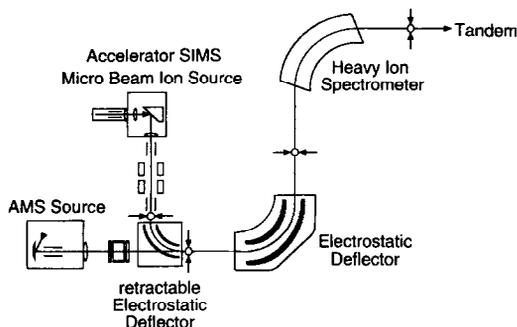


Fig. 4. Part of the low energy side of the accelerator which is used for accelerator SIMS. Since the AMS ion source has practically the same injection beamline as the accelerator SIMS ion source, a pilot beam from the AMS source can be used for optimizing the ion optics of the accelerator system. Switching to the accelerator SIMS measurement is realized by simply inserting a small electrostatic deflector.

is extracted from the AMS source and injected into the accelerator. The transmission is optimized by only adjusting the ion optical parameters of the ion source itself. Thus, the beams of the two different ion sources can be matched to achieve equal beam transport. Now the settings of the magnets are changed to the trace element of interest. An intense ion beam of this element can be produced by sputtering an appropriate sample in the AMS source and the setting of the injection magnet can be optimized. Then, a sample which, in general, does not contain the element of interest is sputtered. With some reduction of the beam intensity this usually yields good counting rates for the ionization detector just from the contamination level in the AMS source. Thus, the magnet on the high energy side of the accelerator can be retuned. In a last step the beam is switched back to the accelerator SIMS source by introducing the electrostatic deflector.

A Si sample with a Cu concentration of about  $3 \times 10^{-9}$  has been analyzed with the technique described above. The sample has yielded a counting rate of 0.05 Hz. It could be shown that the setting of the ion optics as outlined above has been optimum without retuning of the magnets.

The use of a pilot beam makes tuning of the AMS system much easier and avoids the introduction of highly doped materials into the accelerator SIMS sputtering chamber. Therefore, it reduces the risk of contamination. Furthermore, it produces a good reference spectrum of the gas ionization counter.

## 5. Conclusions

The accelerator SIMS setup at PSI/ETH Zurich has been completed and has already been operated in first test measurements. The AMS pulsing system has been shown to be very useful, in particular when an exact determination of the trace element concentration is desired. However, this technique is limited to a certain range of elements.

For the adjustment of the ion optics, the additional use of the AMS ion source has been shown to be very fruitful. It allows fast and easy optimization of all ion optical components. It is thus not necessary to introduce samples with high trace element concentrations into the accelerator SIMS chamber.

The first experiments have also shown that it is very important to have standard samples for the determination of elemental concentrations. An appropriate counting rate from a standard sample would be about 10 Hz. In our setup this corresponds to an atomic concentration of, for example,  $\text{Cl/Si} = 2 \times 10^{-9}$ ,  $\text{Cu/Si} = 10^{-7}$  or  $\text{Fe/Si} = 10^{-5}$ .

Future work will concentrate on the investigation of the performance of our setup. First, the detection limits of various trace elements in Si have to be determined. In a later step, imaging and depth profiling techniques will be applied to the analysis of dopants and contaminants in semiconductors.

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