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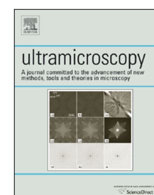

Full length article

Nanometer scale elemental analysis in the helium ion microscope using time of flight spectrometry

N. Klingner [a,b,*], R. Heller [a], G. Hlawacek [a], J. von Borany [a], J. Notte [c], J. Huang [c], S. Facsko [a]

[a] Helmholtz-Zentrum Dresden-Rossendorf, Bautzner Landstraße 400, 01328 Dresden, Germany
[b] Technical University Dresden, 01062 Dresden, Germany
[c] Ion Microscopy Innovation Center at Carl Zeiss Microscopy LLC, One Corporation Way, Peabody, MA 01960, USA

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ABSTRACT

Time of flight backscattering spectrometry (ToF-BS) was successfully implemented in a helium ion microscope (HIM). Its integration introduces the ability to perform laterally resolved elemental analysis as well as elemental depth profiling on the nm scale. A lateral resolution of ≲54 nm and a time resolution of $\Delta t \lesssim 17$ ns$(\Delta t/t \lesssim 5.4\%)$ are achieved. By using the energy of the backscattered particles for contrast generation, we introduce a new imaging method to the HIM allowing direct elemental mapping as well as local spectrometry. In addition laterally resolved time of flight secondary ion mass spectrometry (ToF-SIMS) can be performed with the same setup. Time of flight is implemented by pulsing the primary ion beam. This is achieved in a cost effective and minimal invasive way that does not influence the high resolution capabilities of the microscope when operating in standard secondary electron (SE) imaging mode. This technique can thus be easily adapted to existing devices. The particular implementation of ToF-BS and ToF-SIMS techniques are described, results are presented and advantages, difficulties and limitations of this new techniques are discussed.

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1. Introduction

In the recent past helium ion microscopy [1] has become a mature technique that is best known for its high resolution imaging capabilities. The latest version of these devices, the Zeiss helium ion microscope (model *Orion NanoFab*) (used in this work), is able to operate with He as well as with Ne ions and provides high resolution nano-engineering capabilities [2–4], that so far are unmatched by any other technique. Using neon in the gas field ion source (GFIS) nano-structuring with 2 nm lateral resolution is possible without any metal (Ga) contamination [5,6]. Although exceptional nano-machining and imaging results on insulating and biological samples have been achieved, so far no analytical elemental information can be obtained in the HIM.

Several attempts have been made in the past to obtain analytical information utilizing the nano-sized ion beam available in GFIS microscopes. Early attempts to perform backscattering spectrometry (BS) in a HIM utilized a cooled, windowless silicon drift detector [7]. However, Si particle detectors provide an energy resolution with a low $\Delta E/E$ ratio of just 1:10 or worse. The so obtained BS spectra have been useful only in a limited number of specialized cases. Further, from this attempt it became clear that monolayer sensitivity should in principle be possible [8]. Analyzing the energy distribution of the emitted secondary electrons for elemental analysis has not matured so far [9]. Here, matrix effects and non-linearities in the SE-yield hinder the quantification of the obtained SE energy spectra [10,11]. Recently impressive progress has been made in the development of a dedicated SIMS add-on for the HIM [12,13]. The approach followed by Wirtz et al. [14] will allow high resolution SIMS spectra and mass filtered images with sub-20 nm lateral resolution.

For the elemental analysis by BS several different approaches could be used. For conventional primary ion energies in the range from 100 keV to some MeV various approaches of backscattering energy measurement have been established in the past. Semiconductor detectors are most commonly used and can deliver an energy resolution down to 5.1 keV for 2.25 MeV protons [15] using an in-vacuum preamplifier. Using additional detector cooling an energy resolution of 1.8 keV was reported for 600 keV deuterons [16] and 7 keV for 3.2 MeV He [17]. For low energies such as the ones used in HIM (typically 10–40 keV) energy resolution of 4.5 keV for 25 keV He particles have been reported. These results have been achieved by using a Peltier cooled silicon drift detector [7,8]. Other approaches make use of magnetic [18] or electrostatic energy analyzers which have an excellent energy resolution down

* Corresponding author at: Helmholtz-Zentrum Dresden-Rossendorf, Bautzner Landstraße 400, 01328 Dresden, Germany.
E-mail address: n.klingner@hzdr.de (N. Klingner).

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to $\Delta E/E \leq 0.001$ but are only sensitive to charged particles [19,20] and acquire spectra in a sequential manner.

The fraction of charged, backscattered projectiles for energies below 10 keV decreases rapidly with increasing depth and is below one percent for scattering from depths as low as one nm [21–24]. For energies above 30 keV the charge fraction stays below ten percent [25].

Consequently, for backscattered particle detectors that are sensitive only to charged particles, the overall usefulness is reduced due to the increased sample damage and longer analysis time. The attempt of performing BS in a HIM is connected to a very small beam size and low primary ion energies. It thus is clear that in order to prevent sample damage (by sputtering and/or bubble formation [26–29]) backscattered particle detection has to be sensitive to both backscattered ions and neutrals.

Micro-calorimeters would provide the necessary energy resolution [30] and are sensitive to ions and neutrals but their implementation into the microscope and the decoupling from the heat reservoir of the chamber would require a considerable amount of investigation and engineering work.

The most convenient approach is the application of ToF spectrometry. Performing ToF spectrometry by triggering the start signal from secondary electrons from the sample surface are currently under development for classical Ga focused ion beams [31] as well HIM [32,33]. However, the high number of emitted SEs compared to the rather low cross sections for backscattering lead to a very low coincidence rate and subsequently a poor signal to noise ratio and therefore long measuring times.

Different to previous approaches, here time of flight spectrometry is enabled by pulsing the primary ion beam. We present first analytical results obtained with a combined time of flight backscattering spectrometry and secondary ion mass spectrometry setup. Both techniques utilize the same cost efficient approach, which requires minimal modifications of the system to ensure that the high resolution imaging capabilities are maintained when no analytical information is required. Switching between ToF-BS and standard SE imaging can be performed electronically and requires no mechanical adjustments on the instrument hardware.

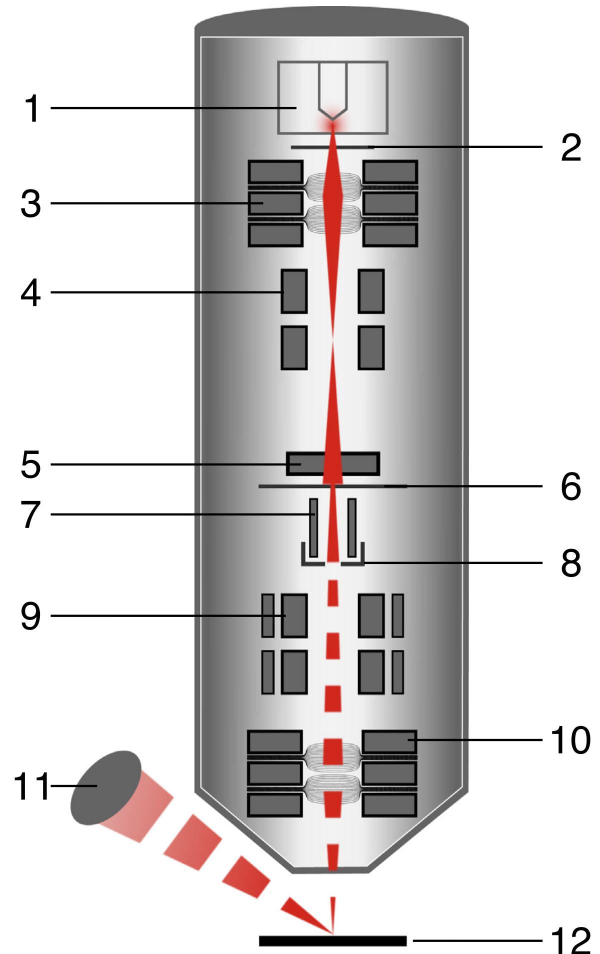

Fig. 1. Simplified scheme of the HIM (not to scale): 1. Source and gas chamber, 2. Extractor, 3. Einzel lens I, 4. Quadrupole, 5. Column isolation valve, 6. Aperture, 7. Blanking unit, 8. Faraday cup, 9. Octopole, 10. Einzel lens II, 11. Micro-channel plate, 12. Sample. The beam path is indicated in red. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this paper.)

2. Experimental

The helium ion microscope delivers primary ion energies from 5 keV to 35 keV, typical ion currents of a few pA and a beam focus below 0.5 nm. Higher currents of up to 150 pA are possible, however only with a larger beam spot and consequently a lower lateral resolution. A scheme showing the major components of the device is presented in Fig. 1.

The start signal for the ToF measurement is created by pulsing the primary ion beam. To retain the excellent imaging capabilities of the microscope no changes have been made to the ion beam column. A newly designed fast pulsing electronics has been added to the column-mounted electronics of the beam blanking unit ((7) in Fig. 1). The new electronics generates fast voltage pulses on both blanking plates that unblank the ion beam from the Faraday cup ((8) in Fig. 1) for a few nanoseconds towards the sample. It is triggered by a standard TTL pulse from a pulse generator with a typical repetition rate of up to 500 kHz. An oscillograph of the voltages on both blanking plates is shown in Fig. 2(a). A rise/fall time of 8 ns equally for both blanking plates was achieved.

The stop signal for our ToF measurements is obtained by detecting the backscattered particles on a micro-channel plate (MCP) referred to as the stop detector in the following ((11) in Fig. 1). It is a chevron stack MCP (model AF2225-A41D, type F1217-01 Hamamatsu Photonics) operated at an amplification voltage of 1800 V. The stop detector is mounted under a backscattering angle of 126° to the primary ion beam and in a distance of 358 mm to the target surface with a solid angle of 10.8 msr. For an increased relative time resolution a second MCP is mounted in a distance of 1023 mm which results in a smaller solid angle of 1.3 msr. The stop signal is amplified by a pre-amplifier (model TA2000B-2, FAST ComTec), the edge detection is done by a constant fraction discriminator (model 2128, FAST ComTec) and the time of flight is measured with a time to amplitude converter (model 2145, Canberra) and digitized by an analog to digital converter (model 7072T, FAST ComTec). Standard spectroscopic equipment (pulse height analysis via a multi channel analyzer) finally reveals the ToF spectrum.

The performance of the ToF setup has been evaluated by direct measurement of the time profile of the pulsed ion beam using a channeltron mounted on the sample stage. The time profile of a 30 keV pulsed He ion beam has been integrated over 2×10^7 pulses and is shown in Fig. 2(b). It can be described by a double error function with a width of 17 ns and a rise/fall time of 1.7 ns.

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