Nuclear Instruments and Methods in Physics Research B 269 (2011) 2175–2179

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Materials analysis and modification at LIPSION – Present state and future developments

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ARTICLE INFO

Article history:
Available online 1 March 2011

Keywords:
LIPSION
Materials analysis
Materials modification
Nuclear nanoprobe

ABSTRACT

The LIPSION laboratory which became operational in 1998 has been improved in numerous ways since that time in order to enhance its capabilities in materials analysis and modification as well as life sciences. This paper summarizes the modifications and improvements made and gives a description of the present state with a detailed list of the technical specifications. The capabilities of LIPSION are illustrated by selected examples from our recent research in materials sciences. Finally a prospect on future developments is given, i.e. the new octupole correction lens and the low noise in-vacuum preamplifier which are expected to further increase the performance of the Leipzig ion nanoprobe.

1. Introduction

Nuclear microprobes (NMPs) are versatile tools that offer a variety of powerful analytical techniques for materials analysis and modification in combination with high spatial resolution [1]. The applications of NMPs in material sciences include quantitative elemental imaging [2,3], the analysis of the structure and defects in crystalline materials [4], the investigation of the electronic properties of semiconductors [5] as well as the modification of a variety of materials in order to create structures [6] or alter physical properties [7]. In some cases, a series of two-dimensional images can even be combined to form three-dimensional tomographic images [8]. This broad range of applications already indicates that nuclear microprobes are far away from being "push-button devices", but are still quite experimental. They are constantly improved to enhance their technical capabilities and to keep up with the demands of today's material sciences. Consequently, the high-energy ion nanoprobe LIPSION which has been operational since 1998 [9,10] has undergone numerous improvements since that time as will be shown here.

This paper describes the present state and experimental capabilities of LIPSION illustrated with some examples of our recent research activities in material sciences. Furthermore, a prospect on future developments is given.

2. Modifications and improvements at LIPSION

The LIPSION laboratory was built completely from scratch. With its special basements, the very stable air-conditioning system, the high brightness 3.5 MV Singletron accelerator and the nanoprobe from MARC Melbourne this laboratory has all ingredients for high-performance and high-resolution microprobe work [9]. Starting operation in 1998, it took a few years to become in-depth familiar with the system, to explore its analytical capabilities and to reveal the weak points every system has in order to make improvements (see Fig. 1). The first major one was the installation of an active compensation of stray magnetic fields [11] in 2002 – the first and up to now only system of this kind used on nuclear microprobes. The compensation significantly reduces the detrimental influence of stray magnetic fields (originating mainly from the tram nearby) on the spatial resolution by keeping constant the magnetic field in the volume that contains the lens system of the microprobe. The upgrade to microDAS in 2005 together with a software developed to scan arbitrary shapes [12] expanded the capabilities of LIPSION by the field of proton beam writing (PBW) [13]. The installation of a new multi-purpose target chamber in 2007 represents another significant modification [14]. It is equipped with a computer-controlled in-vacuum 7-axis stage including a 2-axis eucentric goniometer. The chamber has a removable hood that allows an easy installation of an exit nozzle for external beam applications. Several modifications and improvements followed the new chamber. These are summarized as well in Fig. 1.

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0168-583X/$ - see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.nimb.2011.02.054

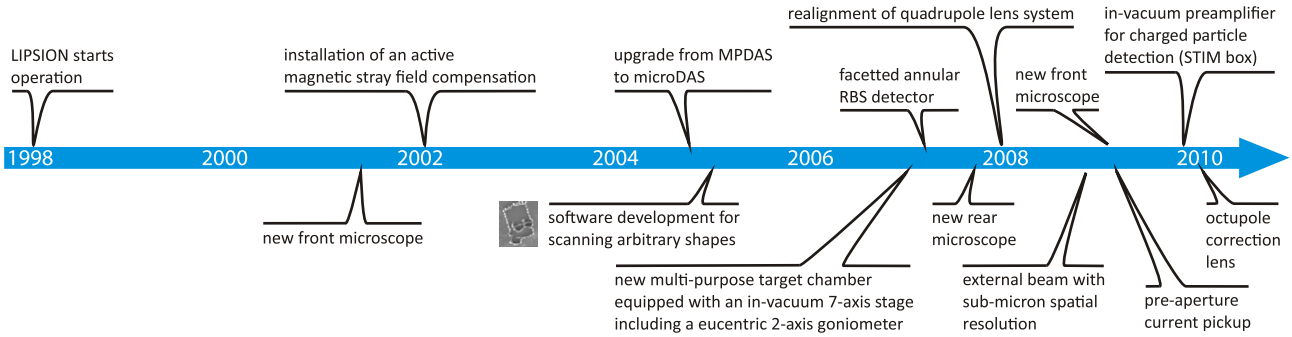

Fig. 1. Modification and improvements of the LIPSION system between 1998 and 2010.

3. Present state and analytical capabilities

A schematic view of the LIPSION laboratory in its present state is shown in Fig. 2 where the main components of the system are labeled. The experimental techniques routinely available are Particle Induced X-ray Emission (PIXE), Rutherford Backscattering Spectrometry (RBS), Secondary Electron detection (SE), Ion Beam Induced Charge (IBIC), and Scanning Transmission Ion Microscopy (STIM). These techniques can be combined with ion channeling using the eucentric goniometer. In addition, PIXE and STIM can be used to obtain 3D-tomographic images by taking a series of 2D-images under different rotation angles. Furthermore, Particle Induced γ-ray Emission (PIGE) can be made available using a dedicated PIGE detector flange.

The sample can be viewed even under ion exposure with a front and rear microscope, respectively. Furthermore, the new front microscope with its large working distance will allow an electrode to be re-installed in front of the RBS detector opposite to the sample that can be biased for secondary electron suppression. Such a system is very important for a reliable measurement of the beam current on target and was already used at LIPSION until 2002 when the front microscope installed at that time made the system ineffective due to geometrical constraints. At present, the accumulated beam charge is calculated from the collected RBS yield.

More technical details of the system are listed in Table 1.

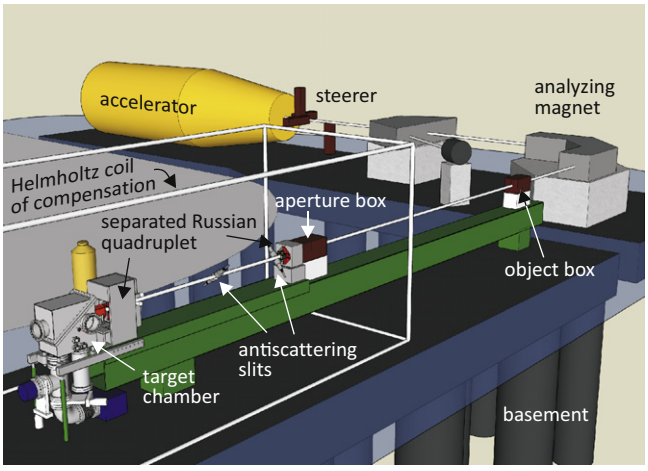

Fig. 2. Schematic view of the LIPSION laboratory.

Today's materials analysis and modification often requires spatial resolutions well below one micron. This can easily be achieved when using low current techniques like STIM or IBIC (neglecting effects like charge diffusion that limit the spatial resolution attainable) or when exposing photoresists in PBW applications. Elemental

Table 1
Technical details of the Leipzig ion nanoprobe in its present state (July 2010).

Beam parameters and ion optics	
Accelerator	3.5 MV Singletron from HVE
Beams	H^+, H_2^+, He^+
Object box	Circular diaphragms + micro-slits; diaphragms with diameters of 5, 10, 20, 30, 50, 100, 200, 300 μm
Aperture box	Circular diaphragms with diameters of 10, 20, 50, 100, 150, 200, 300, 500 μm; pre-aperture current measurement [15]
Focussing system	Separated Russian quadruplet from MARC Melbourne, symmetric demagnification of $D_{x,y} \approx 130$; octupole correction lens in front of third quadrupole (Q3)
Compensation of stray magnetic fields	Active in *x*-, *y*-, *z*-direction using Helmholtz coils from true DC to several kHz, remaining stray field fluctuations ± 10 nT [4]
Beam scanning	Electromagnetic, coils with $N = 1–256$ turns between last quadrupole lens (Q4) and target chamber, maximum scan size: 3.2 × 3.2 mm^2; Murray MA 534 PT 100 transconductance amplifier
Target chamber and its components	
Target chamber	Multi-purpose chamber with detachable hood for external beam applications [6]
Sample stage	in-vacuum computer-controlled 7-axis stage including a 2-axis eucentric goniometer (x, y, z, φ, θ, x', y')
Microscopes	Custom made front and rear microscopes from Thalheim Spezialoptik (Germany)
Charge measurement	Direct pickup from isolated sample holder and/or from a carbon-made faraday cup; no secondary electron suppression at present
External beam	200 nm Si_3N_4 exit window, 2 × 2 mm^2 maximum scan size
Detectors and data acquisition	
PIXE-detector	Canberrra GUL0110 high-purity Germanium (since 2010), 95 mm^2 active area, energy resolution 144 eV at 5.9 keV, retractable with sealed bellow, solid angle up to 150 msr
RBS-detector	Canberra CD-Leipzig1–300 facetted annular PIPS detector, energy resolution 20 keV for 2.25 MeV protons, total solid angle 100 msr (4 × 16.7 msr + 1 × 33.0 msr)
STIM-detector	Hamamatsu S1223 PIN diode, in-vacuum preamplifier based on Amptek A250, energy resolution 5.4 keV for 2.25 MeV protons
SE-detector	Amptektron MD-502 channeltron from Amptek
Data acquisition	microDAS from MARC Melbourne