

## MILESTONE REPORT

# UPGRADE OF THE RBI-AF INFRASTRUCTURE FOR DETECTOR CHARACTERISATION, SEE, MICRO HARDNESS TESTING

### MILESTONE: MS12

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#### Abstract:

This document is related to the implementation of Task 4.2 ‘Micro-beam upgrade at RBI Accelerator Facility’. The aim is to upgrade the two existing ion micro-beam stations by providing a precise motorized positioning system with the beam current control and device under control position monitor, as well as to demonstrate the sample cooling option for the ‘old’ micro-beam station. It has been planned that up to M23 ‘new’ ion micro-beam station will be upgraded (MS12) and that the ‘old’ micro-beam station will be upgraded in the same way before the project end.

AIDAinnova Consortium, 2023

For more information on AIDAinnova, its partners and contributors please see <http://aidainnova.web.cern.ch/>

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## Executive summary

*Task 4.2 targets the upgrade of the accelerator facility for detector characterization, including studying of detector charge collection properties and radiation hardness studies of detector materials at micro-scales. The important milestone, to upgrade the ‘new’ ion micro-beam capabilities has been achieved as planned and described. This includes installation and testing of the precise motorized positioning system with the beam current control and device under control position monitor. In addition to this, we also demonstrate the sample cooling option for the ‘old’ micro-beam station.*

## 1. INTRODUCTION

The Ruđer Bošković Institute runs the Tandem accelerator facility (RBI-AF) used for experiments in nuclear physics and related applications through the Division of Experimental Physics and its Laboratory for Ion Beam Interactions [1]. RBI-AF main applications are materials’ analysis and materials’ modification studies. RBI-AF consists of two electrostatic accelerators with four ion sources and nine end stations (Fig. 1).

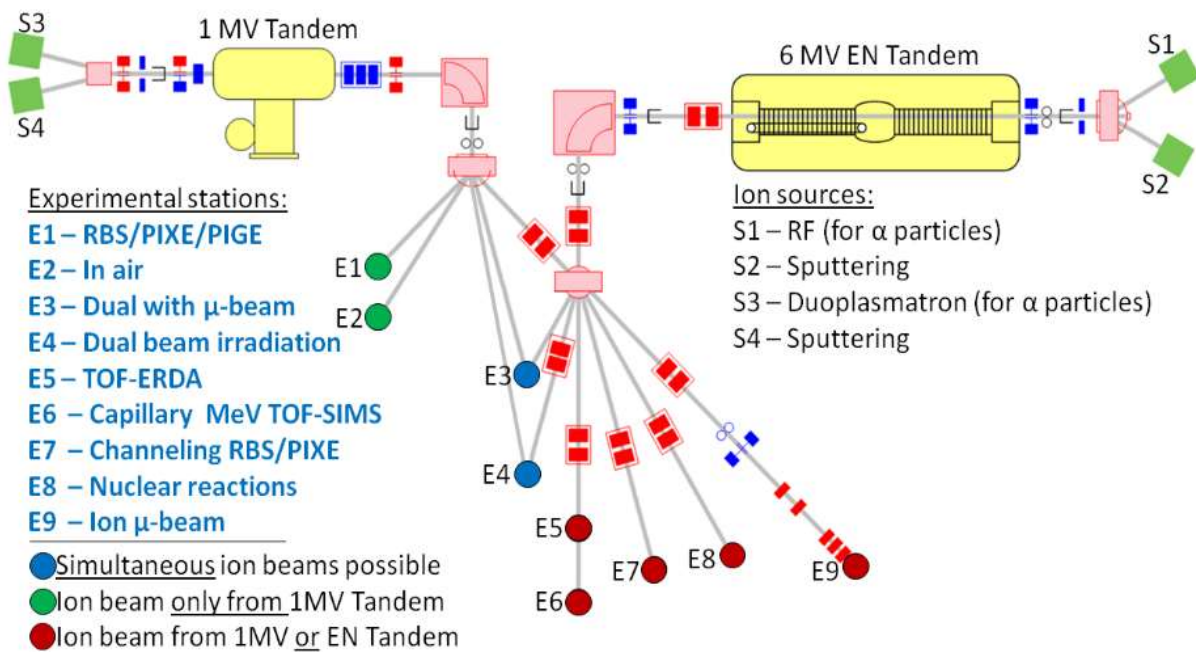


Fig. 1. RBI Tandem Accelerator Facility (RBI-AF) with its two electrostatic accelerators and 9 end stations.

Almost all end-stations, except E1 and E2, are reachable from both accelerators. Two of those, E3 and E4 can accept ion beams from both accelerators simultaneously. Various ions can be accelerated using one of the four available ion sources (Figure 1: S1 to S4). For example, protons can be accelerated to energies between about 100 keV to 10 MeV. In silicon, 1 MeV protons have a range of 16.3  $\mu\text{m}$  and 10 MeV protons a range of 709  $\mu\text{m}$ .

RBI-AF has been regularly used by many international research groups for detector characterization, mainly by exploiting the IBIC (Ion Beam Induced Charge) technique [2] and irradiation by MeV ions for radiation hardness studies [3].

## 2. DETECTOR CHARACTERIZATION AT RBI-AF

In vacuum and in-air IBIC imaging of detector charge collection properties using protons of up to 10 MeV with 1  $\mu\text{m}$  resolution and heavier ions on demand, and/or time resolved IBIC (TIBIC) can be performed. Figure 2 shows ranges of various ions that can be accelerated in silicon.

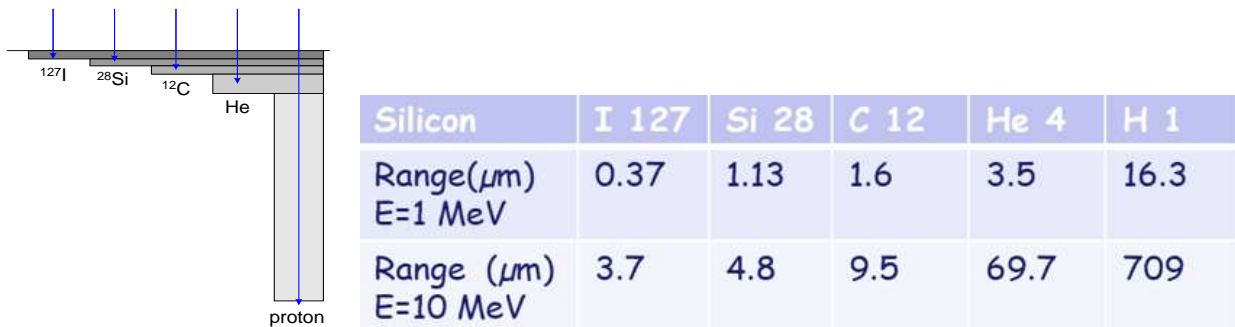


Fig. 2 Range of various accelerated ions in silicon.

Figure 3 shows the sketch of the IBIC experimental setup with focused ion beams. Ion beams focused to micrometer dimensions by quadrupole magnetic lenses are scanned over a sample, i.e. detector under investigation. Detector is powered and its signal is recorded as a function of the ion beam position on the detector. In such a way IBIC charge collection efficiency maps are created. If the detector electrodes are positioned perpendicular to the ion beam than we have frontal IBIC configuration. It is also possible to scan the beam between the electrodes and in that case we talk about lateral IBIC experiment.

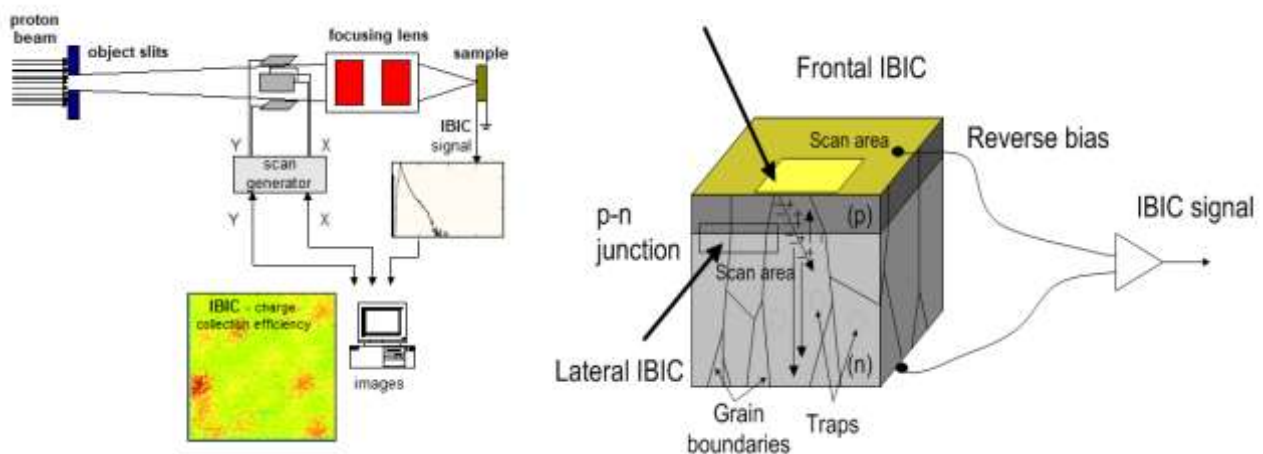
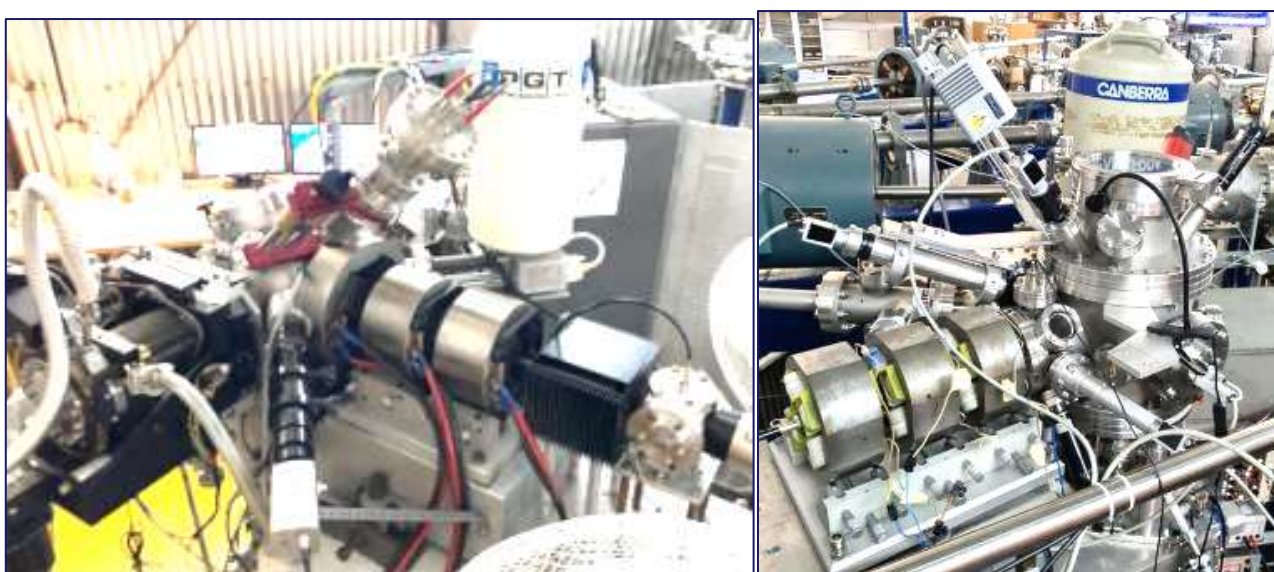


Fig. 3. Left: Experimental setup for IBIC experiments. Right: Explanation of frontal vs lateral IBIC experiments.

For such measurements two ion micro-beam end-stations are available (Figure 1: E3 and E9 and Figure 4).

The left side of Figure 4 shows our "Old" ion micro-beam station [4]. It is equipped with quadrupole triplet magnetic lens that focuses the ion beam to  $\mu\text{m}$  range. Samples can be in vacuum or in-air.

Figure 4 (right) shows our "New" dual beam ion micro-beam station. This station is connected to both accelerators by two beam lines. Therefore simultaneous irradiations are possible at micro-scales. One beam line is equipped with the locally designed and produced quadrupole triplet magnetic lens. The other beam-line is under development and it will enable electrostatic focussing and controlled ion beam scanning over a sample.



*Fig. 4. Left: 'Old' ion micro-beam station. Right: 'New' dual-micro-beam station.*

Regarding radiation hardness studies, this usually includes real-time controlled damaging of small detector areas using protons or heavier ions, including simultaneous or subsequent ion beam analysis. For such studies potential users have at disposal two ion microprobe end-stations and other end stations (Figure 1: E4, E5, E7; and Figure 5) depending on the actual objectives of the proposed work.

Figure 5 on the left shows our dual beam irradiation (DIFU) station [6] for irradiation of areas up to  $2 \times 2 \text{ cm}^2$ . Samples can be irradiated at room temperature or up to 600 C. Central part of Figure 4 shows the Time-of-flight elastic recoil detection analysis (ToF ERDA) end station for thin film analysis which is regularly used for materials science research groups [7]. The right side of Figure 4 shows our ion channelling end-station that is regularly used for irradiation and analysis of crystalline materials [8].

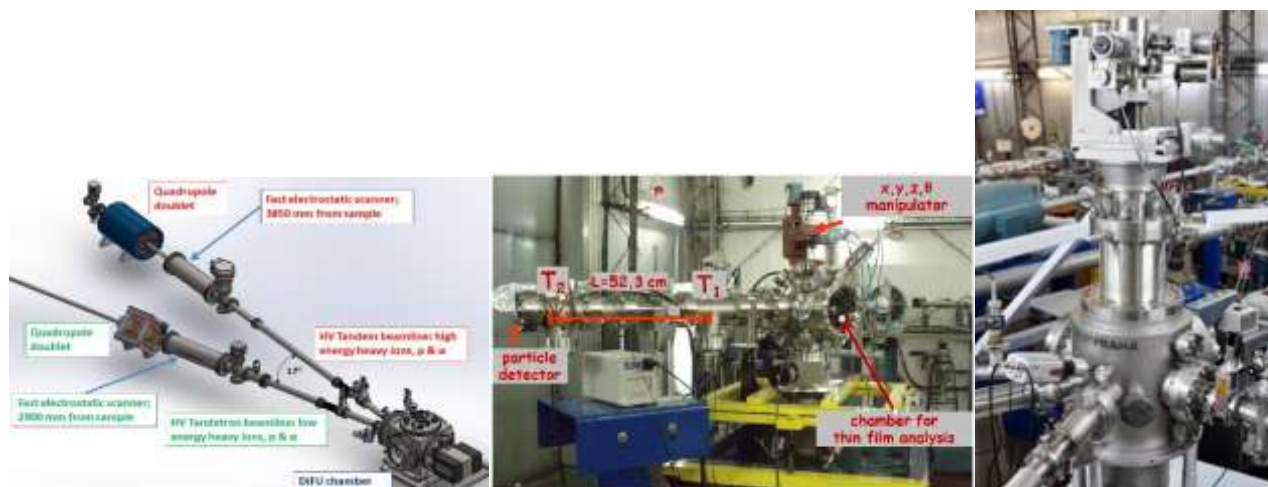


Fig. 5. Left: DIFU station with beam lines. Centre: ToF ERDA. Right: Ion channelling station.

### 3. TASK 4.2 MICRO-BEAM UPGRADE AT THE RBI-AF

#### 3.1. OBJECTIVES OF T4.2

The ‘old’ ion micro-beam station (Figure 2, left) has been in use for about three decades, while the ‘new’ one (Figure 2, right) has been under development during the last several years. At both ion micro-beam stations testing of samples (devices) can be performed in vacuum or in air. At the beginning of the project, sample positioning at both micro-beam stations was manual, not reproducible at the sub-micron scale. Also, the experiments could be performed at room temperature, with a possibility for heating of samples, but cooling was not possible.

The aim of this task is to provide a precise motorized positioning system, cooling system, beam current control and device under control position monitor. Sample cooling is considered only for the old micro-beam station. The goal is also to improve the existing data acquisition and control system and to adapt it to new hardware (motorized sample stages) in order to optimize the facility operation.

Table 1. Present milestone: Upgrade the RBI-AF infrastructure for detector characterization, SEE, micro hardness testing (task 4.2).

Milestone number	Milestone name	Related WP	Due date	Means of verification
MS12	Upgrade the RBI-AF infrastructure for detector characterization, SEE, micro hardness testing	4	M23	Accelerator runs and comparisons with test samples

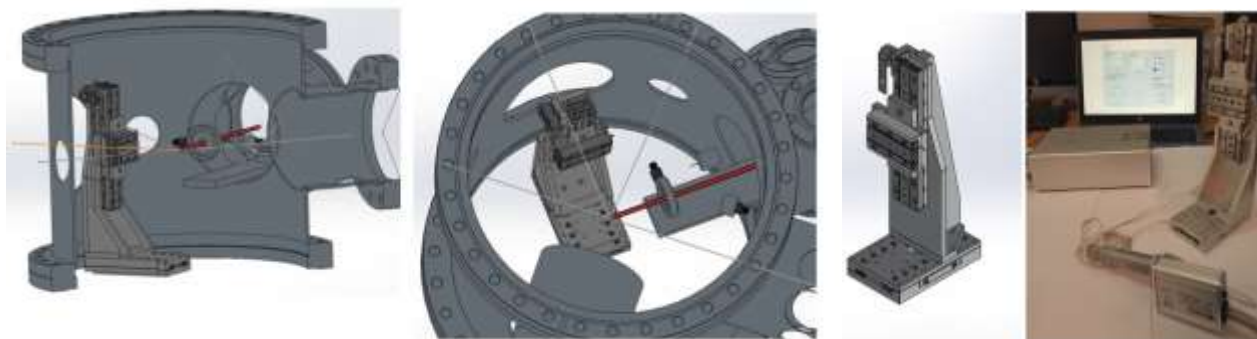
The improved RBI-AF facility will be able to provide the advanced services to the HEP community and beyond (medical physics, hadron therapy, fusion devices, etc.).

It is expected that integrated data acquisition and control system with the motorized sample stages will be available at month 40 of the project. This milestone is set at month 23 (Table 1) when precise motorized positioning system should be procured, installed and tested at the "new" ion micro-beam station.

Once this is achieved, another similar motorized system will be procured, installed and tested at the "old" ion micro-beam station until month 40 of the project. Until then a number of sample cooling options will be tested and applied.

### 3.2. DESIGN AND INSTALLATION OF MOTORIZED SAMPLE STAGES

New precise and motorized sample positioning stage for in-vacuum work at the 'new' ion micro-beam station was designed for the related vacuum chamber (Figure 6). We selected the SmarAct Linear Piezo 3D setup with closed-loop positioning resolution of 1 nm with tens of nm positioning repeatability even over longer travel ranges.



*Fig 6. Design of the vacuum chamber of the 'new' ion micro-beam station and the motorised sample positioning stage. On the right is the procured sample stage with accessories.*

The components for the motorized stage were procured (Figure 6 on the right) and installed. Sample holder is attached on the piezo-stage inside the vacuum chamber (Figure 7). With the new holder, the working range is extended to 100 x 100 x 50 mm. The samples can be positioned at 90 deg and 60 deg with respect to the beam.

Related software for movement of the sample on the piezo-stage has been developed and integrated with the in-house software for data acquisition SPECTOR into the unique integrated software for data acquisition and control (Figure 8).

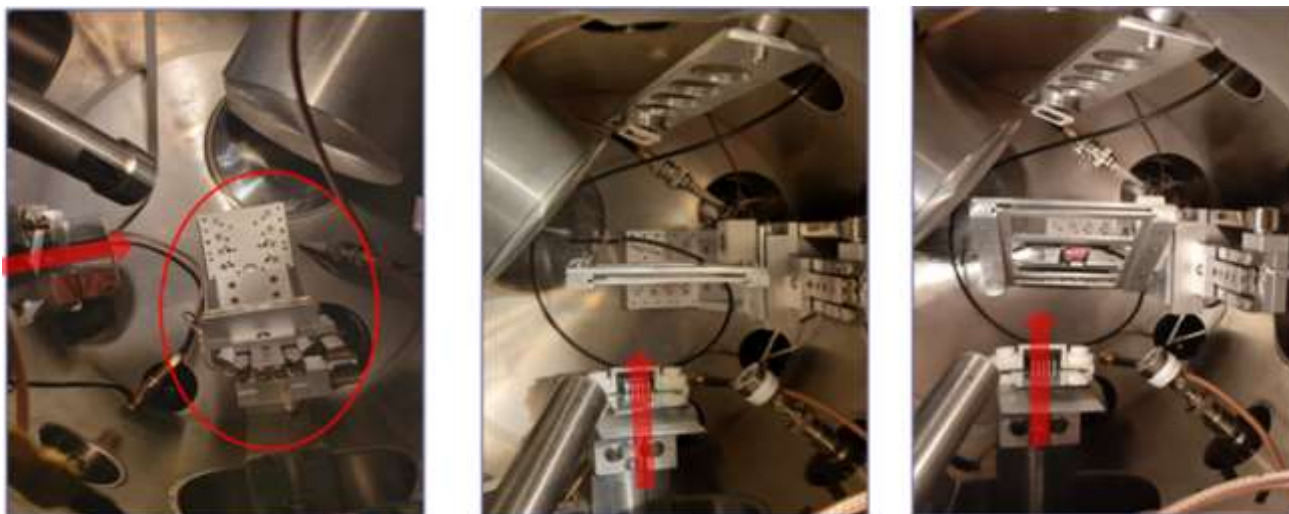


Fig 7. The sample holder attached on the piezostage inside the vacuum chamber of the 'new' ion micro-beam station.

New option to scan the sample in XY plane was added in SPECTOR, extending considerably 2D mapping capabilities (Figure 9). For example, two dimensional mapping of IBIC charge collection efficiency on samples in vacuum has been extended from areas of few mm<sup>2</sup> to few cm<sup>2</sup> in controlled way.

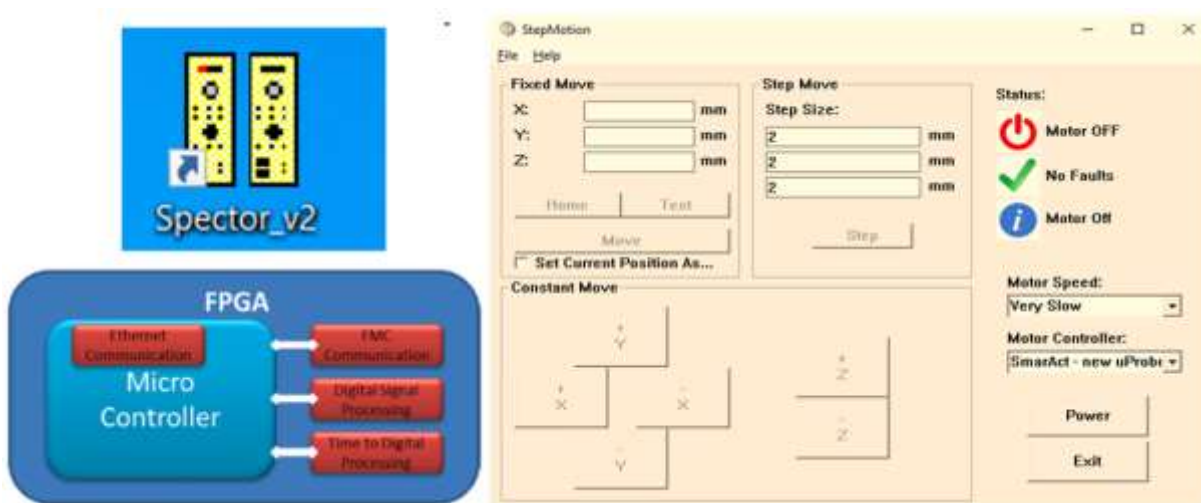


Fig 8. The home-made data acquisition and control software was upgraded to enable the control of the new piezoelectric motorized stage.

A set of tests of the integrated software have been performed, including: tests of different scanning options, tests of collecting multiple spectra, tests of creating multiple 2D spectral arrays, testing of-line analysis of collected experimental data.

Figure 9 demonstrates the test of the scanning capabilities using 2 MeV proton beam on the standard Cu mesh. Measured two dimensional off-axis STIM map of the Cu mesh demonstrates the capability of new option of scanning the sample in front of the beam to obtain high quality images.

Figure 10 shows the result of a test of creating multiple two dimensional (2D) spectral maps on a sample containing various elements. Proton beam x-ray emission intensity maps have been collected from a sample of heterogeneously distributed organic material.

### NEW option to scan the sample in XY plane was added in SPECTOR

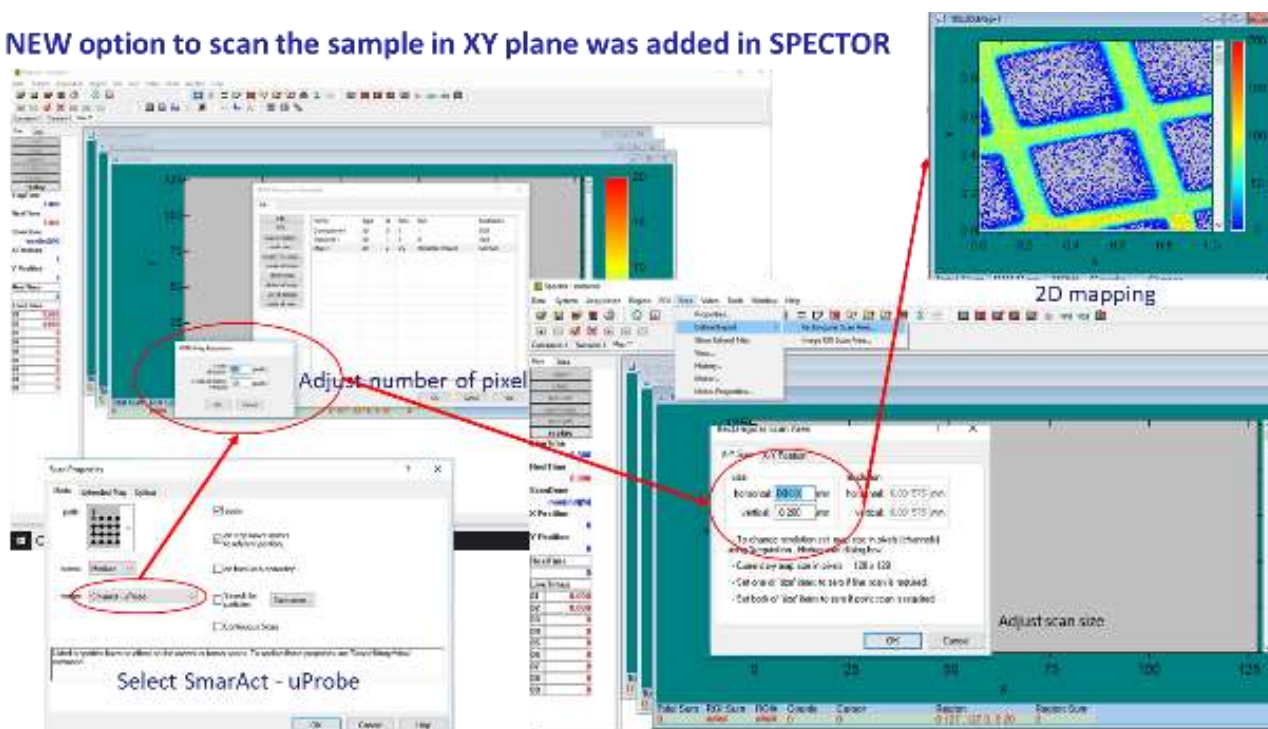


Fig 9. New option added to SPECTOR to enable scanning of samples in XY plane (perpendicular to the ion beam axis) with the example of two dimensional off-axis STIM image of Cu mesh.

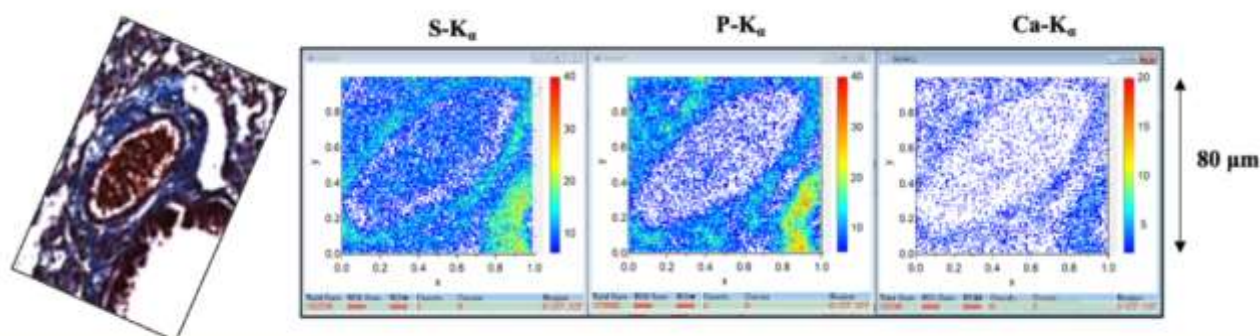


Fig 10. Another example of two dimensional maps of PIXE x-ray intensity maps (Sulphur, phosphorus and calcium) from thin samples containing organic materials.

The above described motorized sample stage is positioned in the center of the main vacuum chamber shown at Figure 6. In this case the samples are positioned in the center of this vacuum chamber. That position corresponds to 'long' focus position, at the same position where the focus of the other beam line is positioned.

However, there is a possibility to position a sample in front of the main vacuum chamber at the so called 'short' focus position, shown at Figure 11. At this short focus, due to the high demagnification, beam spots below  $\mu\text{m}$  can be achieved.

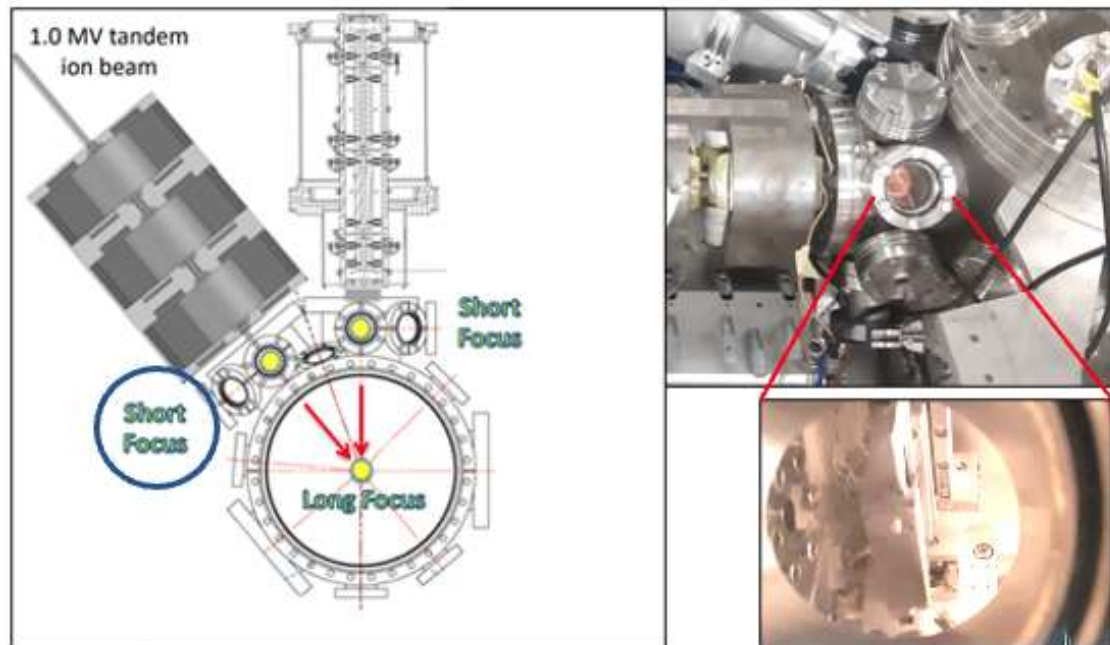


Fig. 11. Two sample positions: 'long' focus in the center of the main vacuum chamber, and 'short' focus in small chamber in front of the magnetic quadrupole lens triplet.

For the sample positioning at the 'short' focus position, another SmarAct motorized piezo stage with 10x10x10 mm working range has been procured, installed and tested. Figure 12 shows tests performed on micro-machined Ni grid with structures down to 400 nm positioned at 'short' focus at the working distance of 70 mm from the lens system.

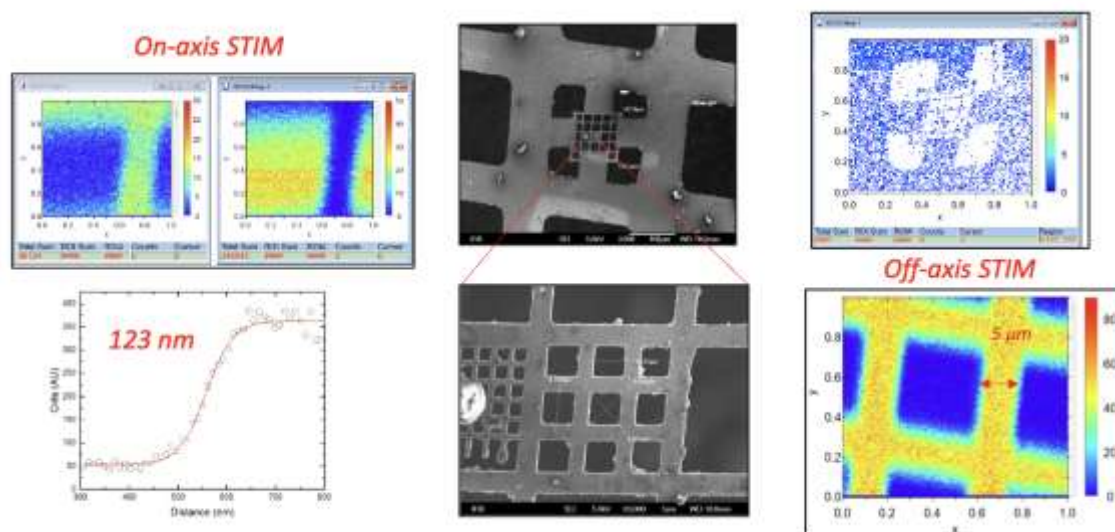
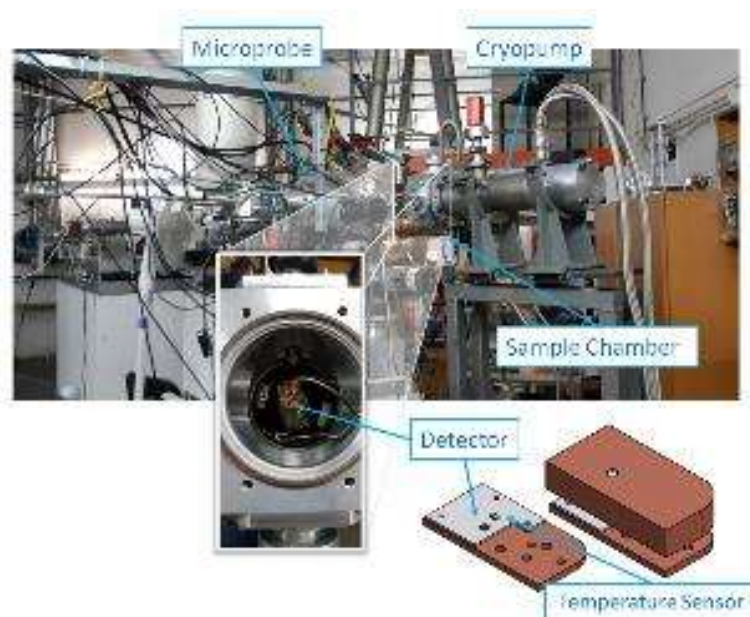


Fig. 12. On-axis and off-axis 2D images of Ni grid structures obtained at the 'short' focus position.

### 3.3. COOLING OPTIONS FOR 'OLD' MICRO-BEAM STATION

Sample cooling has been considered only for the old micro-beam station. Several solutions have been proposed. Two different sample cooling options have been tested: (i) cooling to cryogenic temperatures (Figure 13); and (ii) LN2 cooling (Figure 14).



*Fig 13. Testing of liquid He cooling (IBIC was done on a detector cooled down to 38 K).*

Prototype setup for cryogenic cooling was designed and tested for micro-beam scanning over samples. Temperatures down to 38 K were achieved. Integrated heater for intermediate temperature control was installed and tested. The setup has been already used to study charge collection efficiency from diamond detector at low temperatures [9], a topic of importance for the EUROfusion consortium.



*Fig 14. Testing of passive LN2 cooling (IBIC was done on a detector cooled down to -4 °C).*

In addition, passive LN2 cooling was tested, but this would not be appropriate for majority of practical cases. Second iteration of active cooling system was proposed for (i) water and (ii) liquid nitrogen cooling. Both systems were designed, built and tested. The lowest temperature achieved was  $-40^{\circ}\text{C}$  for cooling with liquid nitrogen.

## **4. CONCLUSIONS**

Planned activities within the AIDAinnova Task 4.2 have been realized as scheduled. The milestone MS12 has been achieved. Two precise motorized positioning systems with the beam current control and device under control position locator for 'long' and 'short' focus positions at the 'new' ion micro-beam station have been designed, related components procured assembled and tested. The existing home made data acquisition and control system SPECTOR has been upgraded to enable the control of the new piezoelectric motorized stage. The setups have been tested and are operational and in use. Testing in various configurations will continue as on-the-job tests during various experiments.

The remaining work also includes the design, procurement, installation and testing of the similar motorized piezo-stage based holder for the 'old' microprobe. This work is in progress. The experience gained to design, install and test the holders for 'new' micro-beam station will be essential.

Regarding the sample cooling, it has been considered only for the 'old' micro-beam station. As planned we have tested cryogenic and LN2 passive cooling.

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## **ANNEX: GLOSSARY**

<b>Acronym</b>	<b>Definition</b>
RBI-AF	Ruđer Bošković Institute Accelerator Facility
IBIC	Ion beam induced current
TRIBIC	Time resolved ion beam induced current
ToF ERDA	Time of flight elastic recoil detection analysis
LN2	Liquid nitrogen
STIM	Scanning Transmission Ion Microscopy