

## HNPS Advances in Nuclear Physics

Vol 26 (2018)

HNPS2018



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doi: [10.12681/hnps.1833](https://doi.org/10.12681/hnps.1833)

### To cite this article:

Theodorou, A., Kotsina, Z., Axiotis, M., & Apostolopoulos, G. (2019). An upgrade of the materials irradiation facility with in-situ electrical resistivity measurement at the Demokritos TANDEM accelerator. *HNPS Advances in Nuclear Physics*, 26, 250–253. <https://doi.org/10.12681/hnps.1833>

## An upgrade of the materials irradiation facility with *in-situ* electrical resistivity measurement at the Demokritos TANDEM accelerator

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**Abstract** As an important part of fusion materials research, evaluation of radiation damage in fusion materials has been emphasized more than a half century. In order to improve our understanding of radiation damage in fusion materials, an upgrade has been performed of the materials irradiation facility IR<sup>2</sup>, which is located at the NCSR “Demokritos” 5.5 MV TANDEM accelerator. The upgraded facility allows irradiation at higher ion beam currents while ensuring that the target temperature remains below 10 K. It provides *in-situ* electrical resistivity measurements on several samples for real-time monitoring of radiation damage as well as *in-situ* post-irradiation annealing up to 300 K. The upgraded IR<sup>2</sup> facility has been successfully employed in radiation damage and recovery studies of metallic materials with applications in fusion research.

**Keywords** fusion, *in-situ* methods, electrical resistivity, proton irradiation, radiation damage

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## INTRODUCTION

One of the key challenges on the roadmap to fusion energy [1] calls for novel and innovative approaches not only in materials development but also in techniques of materials characterization that will provide detailed information on the microscopic processes governing the failure or success of materials under irradiation. An important part of fusion materials research is the assessment of irradiation damage and the study of the thermal evolution of defects. In order to investigate radiation damage, studies of various nuclear materials for fusion applications have been performed successfully at the 5.5 MV TANDEM accelerator of NCSR-Demokritos in Athens, Greece, at the dedicated ion irradiation facility IR<sup>2</sup> [2]. This facility allows ion irradiation of materials at cryogenic temperatures with simultaneous real-time monitoring of radiation damage via *in-situ* electrical resistivity measurements. However, the lowest temperature that could be attained in the IR<sup>2</sup> facility during irradiation was  $\sim 50$  K due to beam heating of the target. This can be a serious drawback for the study of materials such as tungsten, where radiation defects become highly mobile at temperatures well below 50 K and thus cannot be detected and studied.

The upgraded facility consists of a new closed-cycle He cryogenic refrigerator, which reaches a lowest temperature of 3 K and provides 1W of cooling power at 4K. This temperature is sufficient to ensure that the target-material temperature remains below 10 K during irradiation. The cold head of the refrigerator required the design and installation of a new vacuum chamber and the appropriate coupling of the whole setup to the accelerator

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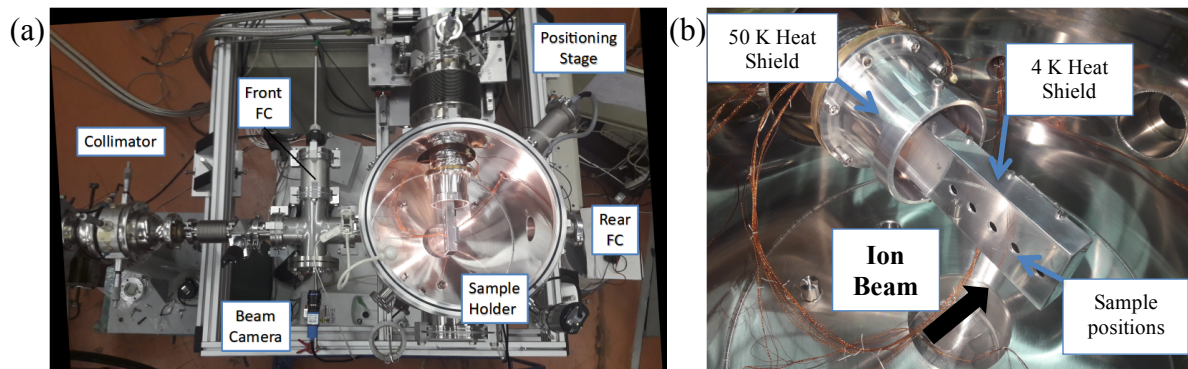
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beam line. The sample holder was designed specifically to allow irradiation of multiple samples and maintain the temperature below 10 K during irradiation. In addition, a suitable software was developed to improve data acquisition and the analysis procedure. The new setup has been successfully completed and tested at the accelerator and irradiations of Al have been performed.

## THE IRRADIATION FACILITY

The irradiation facility IR<sup>2</sup> (“Ion iRradiation with *In-situ* electrical Resistivity measurement”) is located at the 5.5 MV TADEM accelerator of the Institute of Nuclear and Particle Physics of NCSR “Demokritos”. The accelerator can deliver protons, deuterons and light ion beams up to O. After passing through the analyzing and switching magnets, the beam is injected into the line of IR<sup>2</sup>. The line is equipped with an aluminum diffuser foil of 2  $\mu\text{m}$  thickness, which is placed at approximately 2 meters before the target to induce some angular spread and create a more homogeneous beam profile at the sample position. The ion beam is restricted to a 10 x 10 mm<sup>2</sup> square shape by a collimator at 50 cm before the sample. The essential elements of the IR<sup>2</sup> station, shown schematically in Fig 1, are:

- Closed-cycle He cryogenic refrigerator SRDK-408K
- Vacuum system
- Motorized Positioning Stage
- Multiple Faraday Cups
- Beam imaging camera
- Electrical measurements system
- Sample Holder



**Fig 1.** (a) Top view of the upgraded system commissioned at the accelerator lab; (b) sample holder attached to the new 4K cold-head.

Before and after the sample stage, there is a Faraday cup where the ion beam current is measured. A quartz plate can be inserted at 45° angle in the beam path, where the beam generates light by ionoluminescence which is captured by a CCD camera. Thus, a 2-D intensity profile is obtained. The new irradiation target chamber is connected to a dry backing and a turbo pump so a clean high vacuum of 10<sup>-6</sup> mbar can be achieved. The closed-cycle He cryogenic refrigerator SRDK-408K has two temperature stages. A heat shield is connected at the 1<sup>st</sup> stage of 50 K, which encloses the sample holder and protects the samples from thermal radiation. The 2<sup>nd</sup> stage allows a lowest temperature of 3 K and provides 1 W of cooling power at 4 K. A sample holder made of Al6063 alloy is attached at the 2<sup>nd</sup> stage of the cold-head, which provides 5 irradiation positions for samples. The adjustment of the relative

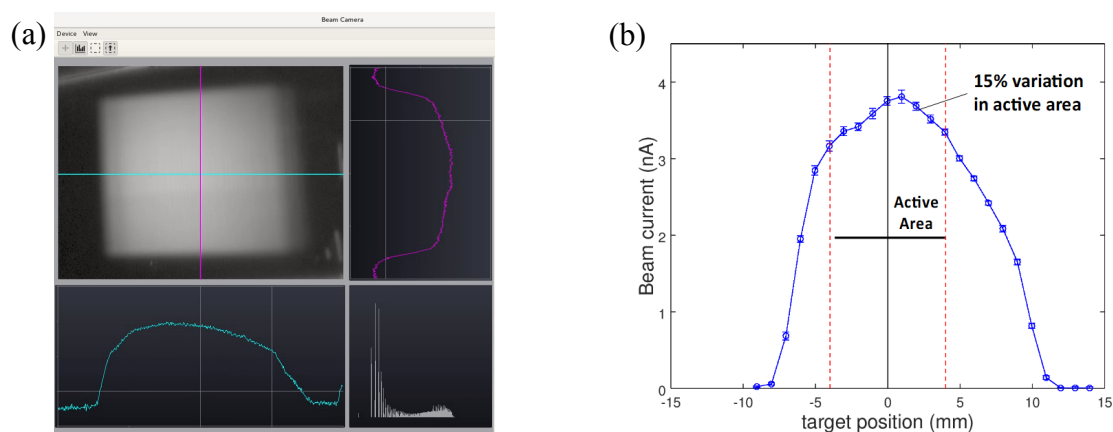
position of cold-head to ion beam is achieved by a motorized positioning stage with a total stroke of 120 mm. The stage is operated by a stepper motor and provides absolute position reading with 1  $\mu\text{m}$  resolution by means of a magnetostrictive sensor. Furthermore, the sample holder is equipped with silicon diode thermometer and a resistive heater for annealing from the cryogenic range up to 300 K. The electrical measurement system consists of a nanovoltmeter, a picoammeter, a precision current source and a versatile scanning system allowing the measurement of several low-level voltage, resistance and current signals with 10 nV, 0.1  $\mu\Omega$ , and 1 pA resolution, respectively.

## EXAMPLES OF THE IR<sup>2</sup> FACILITY IN OPERATION

IR<sup>2</sup> is dedicated to radiation damage studies of fusion materials. The upgraded system ensures that the target sample temperature remains below 10 K during irradiation. This is essential for the study of materials such as W, where radiation defects become highly mobile at such low temperatures. IR<sup>2</sup> allows irradiation, *in-situ* electrical resistivity measurement and post-irradiation annealing of several samples simultaneously. Thus, the upgraded facility offers higher flexibility and efficiency for performing radiation damage studies of materials.

### *Ion beam profile*

A nearly flat lateral ion beam profile is highly desirable for homogeneous irradiation of specimens. The IR<sup>2</sup> facility offers two different ways of monitoring the ion beam profile. To obtain a 2-D beam image, a quartz plate is inserted in the beam path. The resulting ionoluminescence is captured by a CCD camera and a 2-D beam intensity profile is produced. An example profile is shown in Fig. 2(a). The image corresponds to the beam just after the 10 x 10 mm<sup>2</sup> square collimator slit and exhibits a smooth intensity profile in both the horizontal and vertical axes with respect to the beam direction. Another way of checking the beam intensity is by scanning a 2 mm thin slit along the target position. The beam current that passes through the slit is measured by a Faraday cup at the end of the beam line. A typical such measurement is presented in Fig. 2(b) as a function of slit position. It is observed that the beam intensity variation is less than 15% in the active target area of  $\pm 4$  mm.

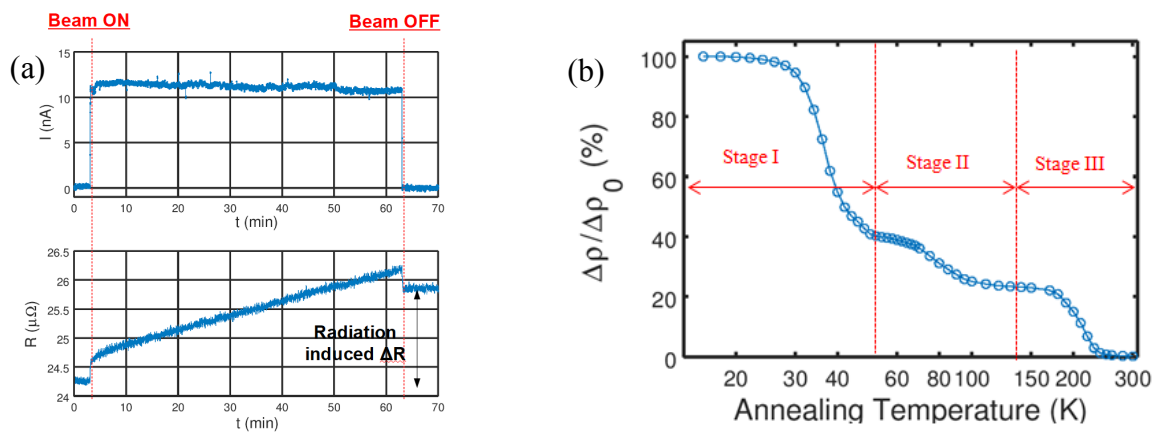


**Fig 2.** (a) 2-D beam profile by CCD camera, (b) 1-D beam profile by beam current measurement

### *Radiation damage production and annealing*

The passage of energetic ions through matter causes atomic displacements leading to the creation of structural defects as vacancies and interstitials. Electrical resistivity is one of the most sensitive methods to the presence of defects and can provide information about their

migration, clustering and dissociation. Irradiation in IR<sup>2</sup> is typically performed at cryogenic temperature so that the generated defects are initially immobile. Fig.3(a) presents traces of the beam current and the increase of the electrical resistance of an Al sample during 60 min irradiation by 5 MeV proton irradiation. Sample temperature was 15 K. The observed increase in resistance is due to the accumulation of defects generated by the protons. After low temperature irradiation and a total resistivity increase  $\Delta\rho_0$  the sample is annealed for 10 min in gradually increasing temperature  $T=15\ldots300$  K. After each annealing step the remaining resistivity increase  $\Delta\rho$  is measured. Fig. 3(b) shows the ratio  $\Delta\rho/\Delta\rho_0$  as a function of annealing temperature. As it is seen,  $\Delta\rho$  decreases gradually to 0 in three distinct temperature ranges or “recovery stages” which are directly related to defect migration, interaction and annihilation. Such “resistivity recovery” experiments are essential for the study of fusion materials because they reveal the kinetics of radiation defects.



**Fig. 3** (a) Beam current and electrical resistivity of Al sample during 5 MeV proton irradiation at temperature of 15 K, (b) Post-irradiation resistivity recovery annealing

## CONCLUSIONS

The IR<sup>2</sup> facility was upgraded successfully and allows ion irradiations at temperature below 10 K, higher ion currents and simultaneous irradiation and measurement of several samples. These improvements extended the range of materials that can be studied and improved the experimental throughput of the facility. IR<sup>2</sup> has been described and its essential elements and specifications were mentioned. The applications of the facility are in the areas of radiation effects and examples of work which can be performed were described.

## ACKNOWLEDGMENT

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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