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Development of the St. Andre Ion Beam Analysis Facility at Notre Dame

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Abstract. A refurbished 3 MV tandem Pelletron® accelerator has been installed in the University of Notre Dame's Nuclear Science Laboratory for the purpose of ion beam analysis applications. A modified Alphatross® ion source provides microAmps of H, He or both ions into the accelerator. Several high-energy beam lines are operational, which allow for internal and *ex vacuo* ion beam analyses. The external beam capability allows for gamma-ray (PIGE) and x-ray (PIXE) analyses to be conducted simultaneously on targets in atmosphere. Rutherford backscattering (RBS) analysis as well as PIXE can also be performed in a 30" scattering chamber. An overview of the new facility, its commissioning experiments and routine operating procedures will be presented together with some examples of ion beam analysis applications performed to date.

INTRODUCTION

The Nuclear Science Laboratory (NSL) at the University of Notre Dame is one of the world's premier laboratories in nuclear astrophysics with a strong emphasis on experimental studies of stellar burning processes. The NSL operates a 10 MV Tandem-(FN)-Pelletron® accelerator[1] and a 5 MV single-ended (5U) Pelletron® at Notre Dame[2], as well as a refurbished 1 MV JN Van de Graaff accelerator installed deep in the Sanford Underground Research Facility in South Dakota[3]. The newest addition to the NSL is a 2000-square foot experimental facility designed for ion beam analysis. The St. Andre Facility is based on a fully refurbished 3 MV tandem electrostatic Pelletron® accelerator with a modified AlphaTross® ion source[4]. Capable of analyzing samples in both vacuum and atmosphere, St. Andre will primarily be used to conduct PIGE, PIXE, and RBS analyses, although fundamental science experiments and some limited irradiations will be available as well. PIXE, PIGE, and RBS are all well developed ion beam analysis techniques for identifying the elemental composition of a sample. Typically, these techniques are performed in vacuum, where the limitation on the rate of sample throughput is set by breaking and reestablishing vacuum. This facility is designed to run many ion beam analyses at slightly higher beam energies ex vacuo, where simultaneous PIXE and PIGE measurements can be used for elemental identification much more rapidly and on targets that do not need to be exposed to vacuum.

This facility will spearhead a new program in applied nuclear science and accelerator applications, most notably in the area of environmental screening and fate and transport studies of

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environmental contaminants. As a University laboratory facility, one of the primary missions of the facility will be to provide educational and training opportunities for a diverse student body. Both graduate and undergraduate students will be involved in the set-up, operation and analysis of experiments performed at St. Andre. This paper presents the capabilities of the new facility, with preliminary results from both commissioning and application experiments.

THE FACILITY

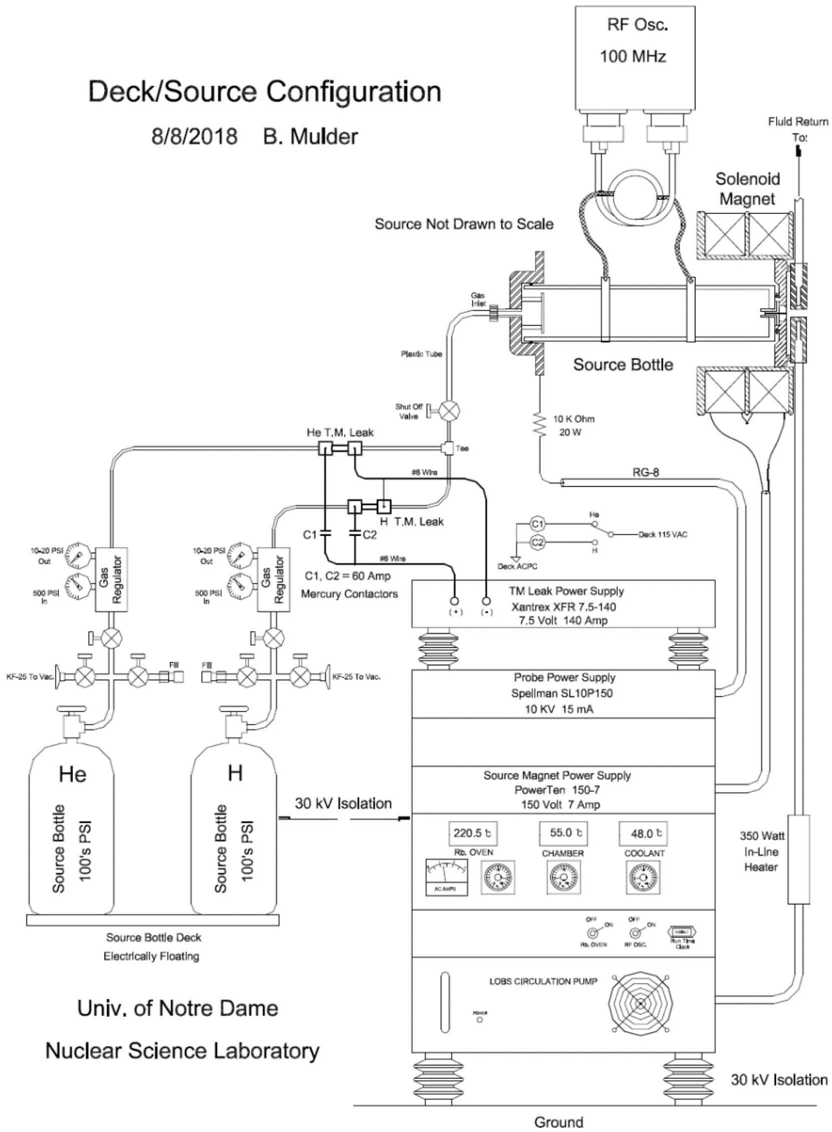
Ion Source

The single ion source is a significantly modified National Electrostatics Corporation (NEC) Alphatross®, which can produce microamperes of either H or He beam. In this system, H_2 or He gas is introduced through a rigid manifold connected to individual thermo-mechanical leaks for each gas. It is possible to introduce a mixed gas species as well, for rapid changes between beam types. The gas pressure in the RF cavity is regulated by the thermo-mechanical leak system, in which the flow of gas is controlled by changing the volume of a metallic valve sleeve attached to the source gas bottle. The volume can be finely and rapidly controlled by adjusting the electrical current across the sleeve, which in turn controls the compression of the seal, and therefore the flow of gas. The source pressure is measured by an ion gauge on the opposite side of the charge exchange cavity. A 100 MHz RF field is used to positively ionize the injected gas in the quartz RF bottle, and a probe bias and a solenoidal electromagnet are used to then force the ions through a cloud of rubidium vapor, where the charge exchange to produce negative ions takes place. All of these components sit on an isolated high-voltage deck operated between 5 – 20 kV, as shown in Figure 1.

The rubidium vapor used in the charge exchange is produced by a band heater on the outside of the oven, configured to uniformly heat the metallic rubidium. The oven is typically heated to 220-240 °C to create rubidium vapor, which is trapped and liquefied onto cooled baffles that surround the charge-exchange chamber. In this source the coolant is preheated to 45-50 °C (above the freezing point of rubidium) via an in-line heater before being transported to the condenser baffles, to prevent rubidium freeze-out. The temperature of the charge-exchange chamber is regulated by a combination of oven heating, coolant temperature regulation, and with direct compressed air cooling, and all three of these have closed-loop control systems to maintain ±1 °C. There are also visible readouts for thermocouples that measure the temperature of the oven, the chamber and the coolant on the front panel.

The ion source is aligned 2° off axis, and is attached to a standard NEC injector assembly with plungeable Faraday cup, an electrostatic velocity selector and steering magnets. Typically, this ion

FIGURE 1. Schematic for the ion source deck for the St. Andre facility.



source can produce 3 – 5 μA of protons, and 5 - 8 μA of alphas on the low-energy cup, and with the modifications as described, can deliver stable beam for 8-hour days on demand.

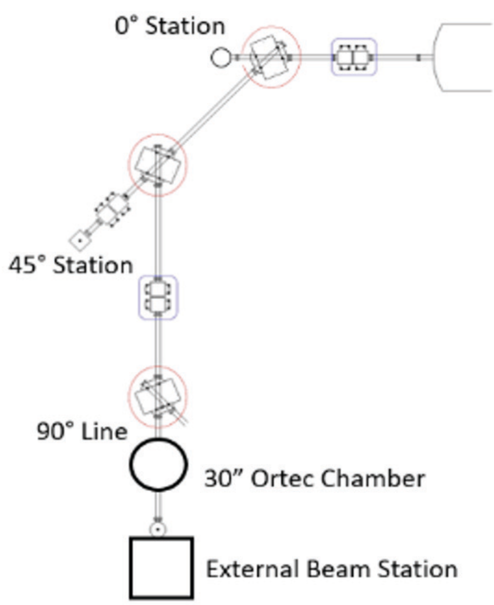
The Accelerator

The tandem pelletron® which is the core of the St. Andre facility is a 3 MV NEC 9SDH Pelletron® accelerator. It was originally a single-ended electron machine that was refurbished by NEC to become a tandem light charged particle accelerator, before being donated to the NSL by Lawrence Livermore National Laboratory (LLNL). Sulfur-hexafluoride is used as the insulating tank gas at 80 PSIG, and the system is equipped with a drying recirculator and a gas recovery system which recycles >95% of the SF_6 in the event of a tank opening. It has two pelletron® chains, both driven from the low-energy end of the tank, and charge exchange in the accelerator is accomplished by means of a series of carbon-foil strippers. There are two stripper mechanisms that each hold 90+ foils that are 6 μg/cm^2 thickness. This particular configuration has an internal 50 kV Einzel lens to focus the ion beams immediately inside the low-energy entrance to the tank. Typically, the beam transmission through the accelerator is ~50% for alphas, and ~10% for protons.

Beam Lines

The St. Andre facility is currently equipped with three beam lines, although there is provision for a fourth future beamline. The high-energy layout is shown schematically in Figure 2.

FIGURE 2. Beamline layout for the St. Andre facility.



One unusual feature of this facility is the use of two 45-degree dipole magnets (designed and fabricated at LLNL) instead of a single analyzing magnet. The stability of the GVM control mode, coupled with the simplicity of the ions being accelerated (only protons and alpha-particles) meant that the ability to control the accelerator via analyzing slits was not necessary. In turn, this meant that an analyzing magnet was not required, which allowed the less expensive, two-dipole option to be feasible.

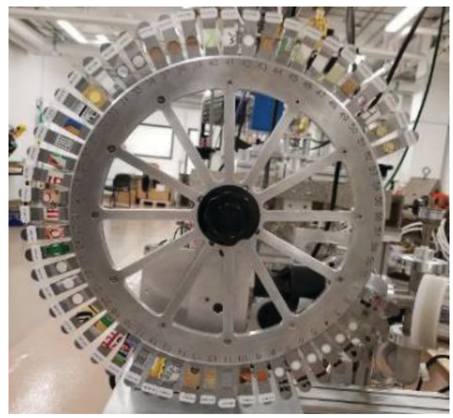
A third 45-degree dipole is installed on the 90-degree line with an option for a fourth (135° line) in the future. Each dipole has both a zero and 45-degree exit port, which allows for a zero-degree irradiation station, which is primarily used as a tuning and diagnostic aid with a quartz viewer and Faraday cup, although some calibration experiments were performed here also. There are two sets of in-line quadrupole doublets for focusing, one immediately after the tank exit, and one on the 90-degree line. With a single quadrupole on the 45-degree line to adjust for beam dispersion in one dimension that occurs after the first 45-degree magnet, which is corrected in the second 45-degree magnet for the 90-degree line.

The 45° beam line is used for *ex vacuo* sample irradiation, primarily with He^{2+} beams, with an isolation valve and thin metal foil window. The 90° beamline is the primary line used for ion beam analysis, as it has a 30" Ortec scattering chamber in place for *in vacuo* measurements of PIXE and RBS, and immediately upstream of the chamber is a plungeable NEC Faraday cup. There is also an NEC beam profile monitor located there, a 1 m beamline extension with a fast-slammer valve at the end, and an exit port with a replaceable 8-micron Kapton® foil (SPEX SamplePrep, Metuchen, NJ). The fast-acting "slammer" valve is wired to a cold-cathode gauge vacuum sensor directly behind the Kapton® foil, in order to protect upstream components in the event of a foil rupture. The Kapton® foil is typically replaced every 24 hours of irradiation as preventative maintenance to prevent foil thickening and embrittlement, and no foil ruptures have occurred to date. Protons and alpha-beams can be extracted at this station for *ex vacuo* PIGE and PIXE analysis. At the 90° beam line Faraday cup, we can typically obtain a stable 50 – 100 particle nA of protons on target, while we can obtain approximately double that for alphas.

There are a set of water-cooled adjustable 4-jaw slits immediately before the first set of high-energy quadrupole magnets, and there are a set of two rotating 3-position collimators in front of the 30" Ortec chamber. Finally, there is a 1 cm diameter tantalum collimator at the exit port of the 90° beam line. The diameter of the beam is typically between 3 and 7 mm at the external target on the 90° line and on the scintillator at 0°. Using the single quadrupole section on the beam at 45° it is possible to achieve a vertically dispersed beam to about 1 cm, while the horizontal dimension remains between 3 – 7 mm.

The external ion beam analysis capability of this facility allows rapid ion beam analysis of targets, and for that purpose a rotating target wheel was built at the 90° end station, that is shown in Figure 3. We use a 60-target wheel that can be mounted by a quick release center screw, and that has a microcontroller that can remotely control the position of the targets radially within 0.1 mm. A camera and scintillator are used to align the beam initially, and then the targets can be stepped sequentially through for routine ion beam analysis.

FIGURE 3. Target wheel at 90° end station.



Detectors

Currently, St. Andre uses separate detectors for each of the various applications. A Canberra high-purity germanium detector (20% relative efficiency, resolution <2keV) is used for detecting gamma rays for PIGE studies. It is Peltier cooled (the Peltier cooling does not introduce any measurable noise to the system), and the lack of liquid nitrogen allows for it to be easily moved from the 90° line to other beam lines as required. While in position at the 90° beam line, it is mounted approximately 2"from the target at a forward angle of 70° to the incident beam. An electrically cooled, Amptek silicon drift detector (resolution ~0.1keV) is also mounted in atmosphere at the 90° beam line, where it is used as an x-ray detector for PIXE studies. It is mounted directly above the beam line, at an angle of 100° with respect to the incident beam, also less than 2" away from the target. An Ortec surface barrier detector is installed in the 30" scattering chamber on the 90° line as a particle detector for RBS studies.

UPGRADES

The St. Andre facility (accelerator and ion source shown in Figure 4) became operational in March 2018 and several months of beam testing and preliminary ion beam analysis experiments have been performed. The first observations using this new facility are very encouraging, and some of the highlights are described here.

The ion source modifications have remarkably improved the ion source stability with respect to other similar ion sources. The use of thermomechanical leaks allows direct electrical control of the gas pressure in the RF plasma bottle with much greater reproducibility and stability than soft-seal valves. In addition, running the Rb oven coolant at temperatures above the melting point of Rb

(~40°C) seems to have solved the periodic clogging of the ion source with solid rubidium. The cooling baffles emerge with barely a dusting of Rb when inspected after a month of use. This modification involved adding a resistive heater to the "coolant loop", replacing the tubing with high-temperature plastic tubing, and adding regulators to the coolant fan, resistive heater and compressed air flow directed at the Rb oven exterior. Standard commercial microcrontrollers regulate all three systems to within ±1 °C. It has also helped to have direct temperature readouts of the oven, charge exchange chamber and the coolant loop to diagnose source conditions. We have also switched to using pure aluminum canals and parts for both H and He beams, rather than switching to tantalum lined aluminum for He beams, which means that we can switch between H and He beams typically within 10 minutes on the ion source. We do erode the aluminum parts more quickly with He beams, but they can be swapped out in under an hour when the beam characteristics warrant replacement, and not venting the chamber to swap canals and boron nitride canal holders has increased the operational lifetime of all pieces. The AlphaTross® ion source has always worked well in terms of ion production, producing many microAmps of both H and He beams, but these modifications have led to greater source stability and a reduction of operational down-time over the first few months of operation. We routinely turn on and turn off the source as needed, and get beam on target within 60 minutes of start-up, and often we have had 8-hour or longer runs where the beam intensity remained stable and within 10% of the initial value all day.

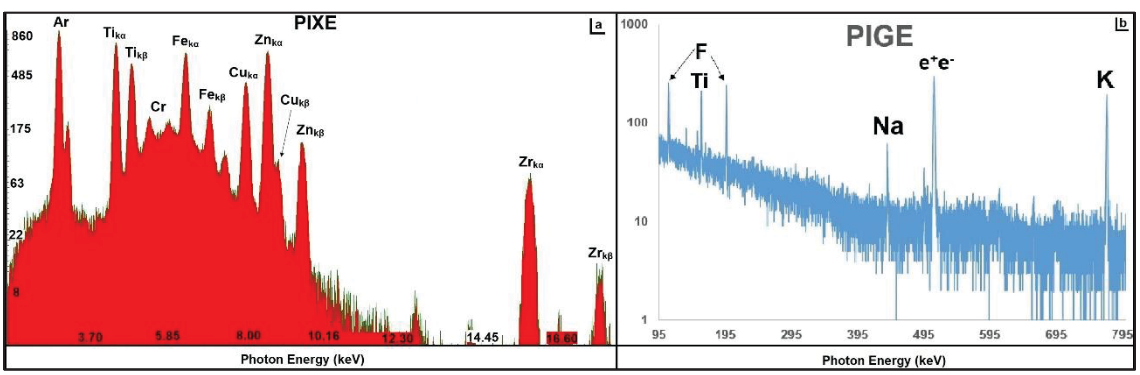
FIGURE 4. The St. Andre modified AlphaTross® ion source and 9SDH accelerator.

Because of the re-configuration of this machine from an electron beam accelerator to a light charged particle accelerator, and the addition of our modified AlphaTross® ion source without additional focusing elements beyond the low-energy velocity selector, the size of the entering beam is quite large for protons, and transmission through the accelerator seems to be low (~10%), despite the internal Einzel lens in the tank. However, the transmission for alphas was immediately 50%, presumably due to better beam divergence from the ion source, so we know the accelerator tube alignment is reasonable, and we typically have too much beam to use for ion beam analysis, so we have not worried about improving the low-energy optics. At a future stage, if a second ion source is added, we may add another focusing objective to the low-energy end to improve hydrogen ion transmission.

PRELIMINARY RESULTS

Preliminary beam energy calibrations were performed with the $^{7}Li(p, n)^{7}Be$ resonance at 1881 keV[5]. These measurements were all performed at the 0° beamline and the terminal voltage was found to be within 35 kV of nominal at 1 MV potential. SRIM[6] calculations have been performed with the exit foils and the estimated beam-in-air distance to calculate the beam energy on target in the *ex vacuo* target stations. Currently, thick cover foils attenuate the x-rays in the Amptek Si detector to be comparable in rate with the Canberra HPGe detector for the measurements of interest, but we plan to incorporate a "funny" filter ([7], and references therein) on the Si detector in order to use the Ar K_{α} lines in air as a real-time measure of the beam intensity on target. While before and after measurements of beam intensity in the high-energy Faraday cup can give very good ion beam analysis results, we think that simultaneous measurement of the argon lines will give even higher precision PIGE and PIXE measurements *ex vacuo*. With the use of a high efficiency germanium detector for PIGE, as well as a beam energy around 4 MeV for protons, it seems possible to measure most of the light elements (Z<30) by PIGE while simultaneously

FIGURE 5. Sample spectra for PIXE (a) and PIGE (b) analyses with select elemental peaks identified. The X-axis is photon energy in keV (cropped to the region of interest). The Y-Axis is counts plotted on a log-scale.



measuring all of the heavy elements (Z> 22) by PIXE *ex vacuo*. The use of *ex vacuo* ion beam analysis has allowed our sample analysis throughput to increase significantly as over 4000 individual samples have been measured in the first three months of operation of the St. Andre facility. Typical analysis for most samples only requires presence/absence determination for given elements (most commonly fluorine), which is ideal for rapid PIGE and PIXE measurements. Example spectra (from a paper sample) are presented with elemental identification in Figure 5.

CONCLUSIONS

A new ion beam analysis facility (St. Andre) has been installed and is operating at the NSL at the University of Notre Dame. It is distinguished by a 3 MV terminal voltage that can accelerate beams H^+ and He^{2+} ions delivered by a modified AlphaTross® ion source. It produces ~ 100 nA of beam on target routinely and many of the analyses are performed *ex vacuo*, which allow for a rapid sample processing for PIGE and PIXE measurements. The facility is designed for environmental ion beam analyses, but irradiation facilities are in place as well. The facility is also designed to be operated by both undergraduate and graduate students as part of the educational mission of the NSL, and standard operating procedures have been developed.

ACKNOWLEDGEMENTS

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