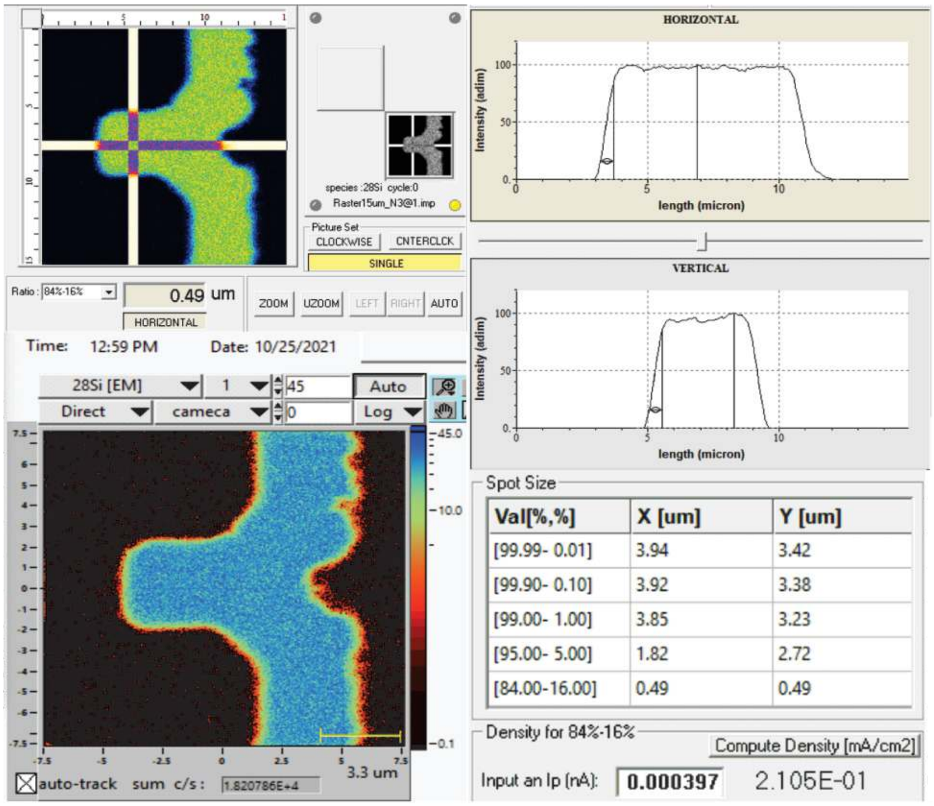

Figure 3: Determination of beam diameter of Cs+ primary beam.

Various Isotopic analyses were carried out to determine the internal and external reproducibility of the measurements. Oxygen and silicon isotopic analyses were done in quartz samples and magnesium isotope analysis was done in olivine samples. Silicon isotopic ratios for 12 spots are shown in figure 4. The internal and external reproducibilities were found to be < 0.05 % for all the isotopic analysis for 12-14 spots having 10 cycles of 4 second integration time each.

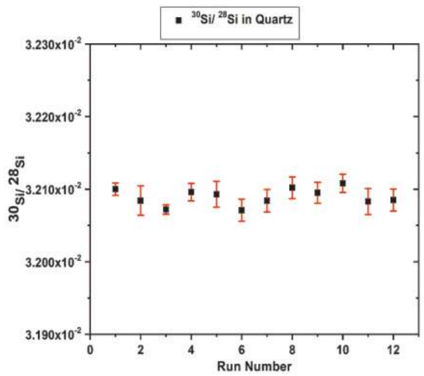

Figure 4: Silicon isotope analysis in quartz.

A Comparison of Zircon dating from two different mass spectrometric techniques was carried out. The dating of zircon samples was done with LA-HR-ICPMS facility at IUAC followed by SIMS analysis. An agreement is found between all the ages within the error bar. For grain #4, the mismatch between the ages may be due to different rims of the grain. The CL images of the zircons with the ages from the two techniques are shown in figure 5.

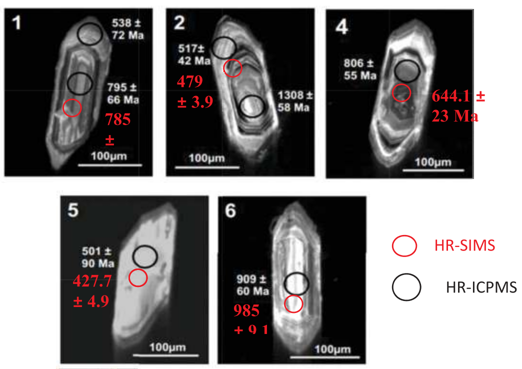

Figure 5: CL images of the grains chosen for the study.

1.4.2.2 Femto second Laser Ablated High Resolution Inductively Coupled Plasma Spectrometry (LA-HR-ICP-MS)

HR-ICP-MS (model: Thermo Scientific, Element XR) coupled with femto second Laser Ablation (model: Teledyne) is now an extensively used instrument for U-Pb dating and isotopic ratio measurements. In laser mode, the sample is exposed to laser which ablates it and converts it into aerosol. The sample is then fed to a plasma through helium. The advantage of HR-ICPMS over conventional ICP-MS is that it uses magnetic sector Single-Collector (SC) Mass Spectrometer (MS) to measure isotopic ratios of elements. The primary application of HR-ICPMS is to measure the isotopic ratios of elements used in geochronologic radiogenic isotopic studies. The LA-HR-ICP-MS has significantly contributed to understand the crustal evolution of the Indian Craton and the different stages of Himalayan orogeny.

Mass calibration is very important to control any shift in the masses of elements and was done at regular intervals with 'tune solution'. For laser mode, tuning was done using the NIST standard. Tuning sets up the instrument parameters (such as sample gas, torch position etc.) in such a way that maximum counts per second can be achieved for the samples. Several standard reference - Zircons such as Plesovice, Temora, 91500 and FC1 were used for optimization of LA-HR-ICPMS. In solution mode ~400 samples (unknown + standards) were measured. Samples were provided by BARC, Mumbai and $^{176}Lu/^{175}Lu$ were measured. In laser mode ~600 grains were ablated for U-Pb dating of Zircons. The pre-Himalayan granitoid bodies of the Chhota Shigri area, Himachal Pradesh were studied. A systematic zircon isotopic study could determine the timing and nature of the magmatism in the region. The study will be helpful in reconstructing the pre-Himalayan setting, and probable source of the pre-Himalayan rocks. Evidence of the involvement of ancient Indian crustal components in the generation of these granitoid bodies may be verified from this study.

1.4.2.3 Quadrupole- Inductively Coupled Plasma Spectrometry (Q-ICPMS)

The Q-ICPMS instrument is a very efficient and convenient tool for elemental analysis at ppb level. ICP-MS technique has been useful in quantitative analysis of trace and ultra-trace elements in samples from all sources like geographic, environmental, biological etc. Trace element analysis of surface and ground water is a useful tool to assess the quality of water and pollution level. Elemental analysis of water/soil can be helpful in identifying the potential cause of local health problems. In addition, it becomes possible to identify the source of pollution/contamination by examining the samples from different sources. Elemental analysis is also helpful in the assessment of different remediating techniques used for removal of heavy/toxic metal load near landfill sites. This will help in improvement of living conditions nearby landfill areas and will decrease level of toxic metals in ground water due to leaching from landfill waste. ICP-MS technique can measure metal concentrations as low as 0.1 ppb.

Figure 6: Q-ICPMS instrument with auto sampler.

At IUAC, Q-ICPMS system equipped with auto-sampler was installed in 2018 and is being used regularly since then. During March 2021 – February 2022, this facility was used for analysis of about 2600 samples by 20 users from different institutions. In addition, the facility was also used for precise estimation of Be and Al in quartzite samples as partial requirement for AMS measurements for surface exposure studies. The major thrust areas of research include:

- Environmental studies- Soil and water pollution.
- Heavy metal concentration in soil, water and plants
- Earth Sciences-Soil profiling
- Water quality assessment and human health risk association
- Micronutrients in Human health and pre-term births

1.4.2.4 Field Emission–Scanning Electron Microscope (FE-SEM)

The JEOL Field Emission–Scanning Electron Microscope (FE-SEM, JSM-7610F) installed in our laboratory at IUAC is a versatile high resolution scanning electron microscope as shown in figure 7. Apart from the high-resolution surface imaging using Secondary and Backscattered electrons detection, this machine has analytical capabilities such as Energy dispersive X-ray spectrometer (EDS) for elemental analysis, Electron backscattered diffraction (EBSD) for crystalline information and *Cathodoluminescence (CL)* for optical analysis of the samples.

For coating of non-conductive samples, we have facility of two compact and automatic rotary pumped coaters (Carbon (EC-32010CC) and Platinum sputter (JEC-3000FC)) as shown in figure 8. The FE-SEM with CL facility competently performed all-round the year except EDS and EBSD. More than 30 users from 17 Universities/Institutes all over India have utilized the facility. Following types of samples were analysed from different fields for surface topography.

- Geological: Quartz and Feldspar grains, petrogensesis of the glass for better evaluation of magma chamber present, *CL of Zircon grains from North-East region and Chhattisgarh etc.*
- Materials Science: Thin films (pristine and doped CdS etc.) and powders (WSe2, NiO, ZnO/CNT nanoparticles etc.) of different materials, cross-sectional (AlN and GaN nanotube structure grown on sapphire) and pellets of $LaCoO_3)_x$- $(Na_{0.5}Bi_{0.5}TiO_3$ made by PVA
- Nuclear target samples: ^{156}Gd, ^{158}Gd, ^{nat}Gd, ^{109}Ag, ^{107}Ag
- Atmospheric Sciences: Aerosols PM_{10} collected on quartz filter from different region in NCR-Delhi (physio-chemical process, source apportionment of PM_{10} and average mass concentration along with morphology was carried out in this study)

Maintenance

- Colour CL (Delmic make) detector is newly installed and RGB mode of detector is under optimization.
- The seal at pivot of the Rotary pump connected to the Turbo pump was not screwed properly and oil had leaked. It was repaired.
- Penning gauge was not reading the vacuum pressure. This was stopping the electron beam from the gun to be turned on due to vacuum interlock. The penning gauge was inspected by Vacuum group at IUAC and maintenance of the gauge was performed after which it was working fine.

Figure 7: FE-SEM (JEOL-JSM-7610F).

Figure 8: Carbon and Platinum Coater.

1.4.2.5 Wavelength dispersive X-rays fluorescence spectrometer (WD- XRF)

Wavelength dispersive X-rays fluorescence spectrometer has been installed and operational since 2018. A fuse bead machine, pellet press and vibratory cup mill are being utilized for the sample preparation. A new calibration is made for major oxides and trace elements of Granites samples. Depending on the silica content of the standards, two different applications were made as described below:

High range (Silica): 72.30 to 76.83 %

Medium range (Silica): 63.34 to 67.77 %

High range silica applications was made with standards JG-2, JG-1a, JR-2, JR-3, CH-22, GeoPT-21, TBG-9, TBG-10, DGH/9, RGM-2.

Medium range silica application was made with standards JG-3, MKM-4, GeoPT-8, TBG-7, GD-14, SARM-48.

Trace element application was made using GD-14, JR-2, JR-3, JG-1a, JG-3, NIMG, SARM-48, GeoPT-8, GeoPT-21, RGM-2.

The facility is being utilized by users from the field of geology, environmental science, nuclear physics, etc. for the study of:

- Paleoclimatic reconstruction.
- Pedogenesis.
- Groundwater potential zones.
- Terrestrial analogue study.
- Genesis of rocks.
- Chemical effect or shift in emitted X-ray lines of different metal complexes.

From April 2021 to March 2022, XRF measurements of 115 samples from 1 user from 1 institute have been done.

Maintenances related to XRF

- The X-ray tube was found off and the chiller showed fault. This was because chiller was not working properly. Chiller was repaired with the help of AC group.
- XRF system was not turning on, there was a problem in switching on the power. There was a problem with the power supply SMPS LPQ-172. The control board and turret motor were replaced with newer ones, as shown in figure 9.
- The screws of pellet pressing machine were found to be broken. The machine was repaired with the help of IUAC workshop.

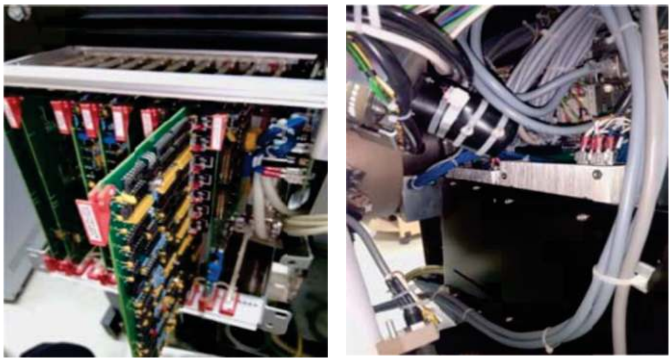

Figure 9: Maintenance related to WD-XRF.

1.4.2.6 X-Ray Diffractometer (XRD)

The X-ray diffractometer has been operational since 2017 and being utilized routinely by users for the characterization of their samples.

Powder XRD method is used to study different types of samples:

geological and environmental; Earth surface process, paleoclimatic study etc.

Materials science: Determination of different phases along with calculation of stress, crystallinity etc.

Nuclear target: Study of formed phases.

Currency notes: Originality check.

From April-2021 to March-2022, 672 samples of 34 users from 12 different universities and 5 institutes, were measured using XRD.

Maintenance related to XRD during the year (2021-2022) is listed below:

Dryer of chiller was choked, which was changed but the X-ray tube didn't turn on. After repairing, water flow was also adjusted and then the X-ray tube could be turned on and the system started working.

1.4.2.7 Laboratory magnetic barrier separator

Most of the minerals fall under three categories of magnetic properties; Ferromagnetic, Paramagnetic and Diamagnetic minerals. Magnetic barrier separator uses magnetic field and gravitational field to separate a particular mineral from the mineral mixture. The magnetic separator has a large electromagnet through which mineral mixtures is passed on an inclined metal plate. By varying the magnetic field value and/or the slope of the metal plate the minerals of different magnetic properties can be separated from one another. The system was commissioned in June 2019 and about 134 samples have been processed for quartz separation for the purpose of CRN dating.

1.4.2.8 Jaw Crusher, Vibratory disc mill and Sieve shaker

These instruments were commissioned in July 2019 and are used for physical processing of rock to convert them in required grain sizes of powder. More than 350 rock samples have been processed for the purpose of CRN, ICPMS and XRF analysis.

1.5 LOW ENERGY ION BEAM FACILITY (LEIBF)

Pravin Kumar and Ambuj Tripathi

1.5.1 Operation

The overall annual performance of 10 GHz Electron Cyclotron Resonance Ion Source (ECRIS) based Low Energy Ion Beam Facility (LEIBF) [1-3] in the academic year 2021-22 is depicted in a pie chart below. The failure of air-conditioning in the ion source room and unexpected delay of about five and half months in the installation of new units (3 instead of 2) caused dead time to be very high (~ 42%). In the second phase of COVID-19 pandemic, the lockdown was declared from April to May 2021 and the user experiments could not be done in fulfillment of standard operating procedures (SOP). From the first week of November 2021 onwards, the LEIBF was made available for conducting user experiments. During the period April 2021 to March 2022, the beam time of 65 shifts (12 user experiments) had been utilized.

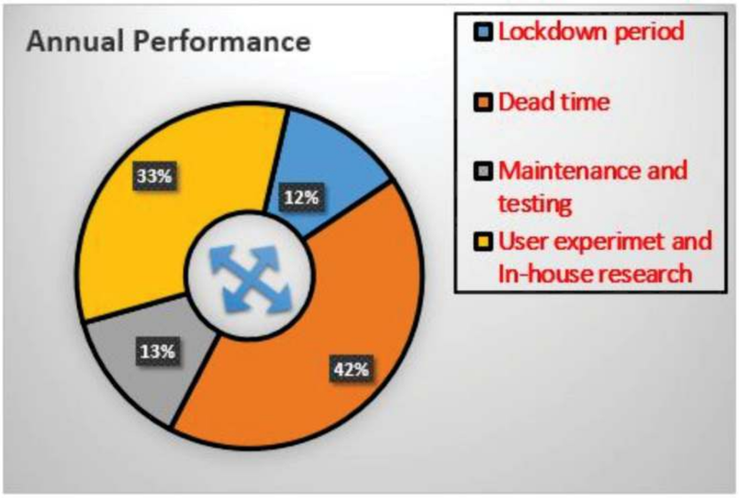

Figure 1: Overall annual performance of LEIBF at a glance.

1.5.2 Maintenance

The important maintenance works carried out in the academic year 2021-22 are as follows:

- The installation of three split AC units in the ion source. The additional one unit was kept for standby.
- The cleaning of vacuum pumps installed in pre-analysis section followed by leak detection tests
- The installation of flow meter with proper interlocks to avoid rusting of the electronic components of water cooled high power magnetic devices
- The voltage multiplier stacks of 400 kV high voltage power supply were thoroughly cleaned using de-ionized water to ensure greater beam stability during regular user experiments
- The scheduled maintenance by various support labs to confirm proper functioning of beam transport system, vacuum components and remote operation of the LEIBF.

Figure 2: (a) The water flow meter installed in the beam line. (b) 3 AC units installed in the ion source room.

1.5.3 In-house research activities

In regards to in-house research programmes, the objective has been to put continuous efforts in exploring the best performance of all-permanent-magnet 10 GHz ECRIS and look for the feasibility of new experiments with the ion beams produced in this course of exercise. The development of high intensity of highly charged ions [4, 5] is the main aim of all researchers working with ECRIS across the globe. To the best of our knowledge, the charge state distributions of C and O influenced by internal as well external mixing (as shown in figure 3) have been studied first time. For this purpose, the CO_2 gas was injected to produce the main plasma and additional helium gas injection was employed for the external mixing of main CO_2 plasma. The helium gas mixing was done at two levels i.e. 50 and 80%. From the charge state distributions of C and O, the mixing effect is very much evident. However, in comparison to helium mixing at 80% level, the helium mixing at 50% level is more effective for both, C and O. Further, the helium gas mixing caused +7 charge state of O to populate in the plasma. Furthermore, the 40 keV C beam was implanted in thin films of Cu deposited on Si/SiO_2 at various fluences to demonstrate the fabrication of a few layers of uniform graphene on Cu. The Raman measurements of the annealed and C ion beam implanted samples are encouraging. However, the analysis of measurements suggested re-optimizing the annealing temperature, heating and cooling rates, ion fluences, etc.

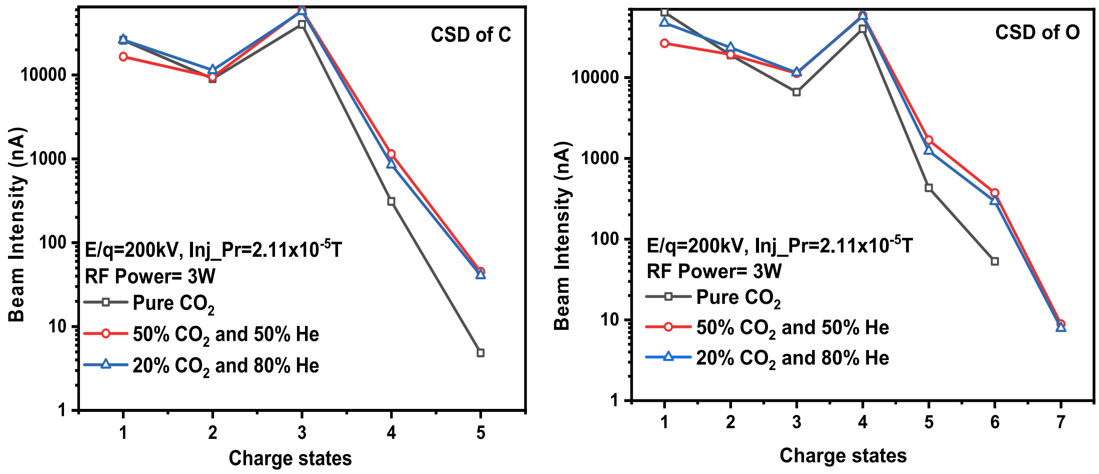

Figure 3: The charge state distribution for C and O with helium gas mixing in plasma.

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1.6 LOW ENERGY NEGATIVE ION IMPLANTER FACILITY

Ksh. Devarani Devi, GR Umapathy, Sunil Ojha, Deeksha Khandelwal, Rajveer Sharma, G Raturi, Jawant Singh, Pranav Singh, Suraj Kumar, Mohan Nishal, N S Panwar, M P Singh, Rakesh Kumar, Jagdish Prasad, Chandra Pal, Ashok Kothari, PBarua, Deepak Kumar Munda, Kundan Singh, VPPatel, M Sota, Ashish Sharma, B.K. Sahu, R.K. Gujar, Mukesh Kumar, S Gargari and S Chopra

1.6.1 Operation

The facility was operational round the year but was hindered by few major breakdowns and COVID-19 Pandemic. In mid-March 2021, the general purpose (GP) accelerating tubes were not holding even moderate voltages; and the cathode was observed to be drawing high current. The breakdown maintenances could not be taken up right away due to onset of the second wave of COVID -19 pandemic and IUAC had to follow the strict standard operating procedure (SOP) guidelines. The normal operation and stability of the accelerator system with its highest acceleration voltage, 200KV was achieved after performing maintenances and conditioning of the machine. The ion beam delivery started from October 2021and since then the performance of the implanter accelerator has been excellent. The operational statistics of the facility is listed below:

Total no. of users: 18

Total time utilized for implantation=448 Hrs.

Total number of samples implanted=581

Ion beams delivered: ^{7}Li, ^{12}C, ^{16}O, ^{28}Si, ^{31}P, ^{40}MgO, ^{56}Fe, ^{58}Ni, ^{59}Co, ^{74}Ge, ^{107}Ag, ^{130}Te and ^{197}Au

Energy range: 30 to 200 KeV

Ion Fluence range: $1x10^{13}$ to $5x10^{16}$ ions/cm^2

Few experiments utilized tilt angle to reduce the depth of ion implantations.

1.6.2 Maintenances

The facility's performance was down primarily due to electrical shorting of the ceramic insulation gaps of general purpose accelerating (GP) tubes as well as cathode voltage fluctuations.Thus, it was decided to bring the negative ion implanter accelerator back in normal operation with improved stability. The following maintenances of the devices listed below were performed:

GP tubes: All the three accelerating tubes were disconnected from the injector beam line. The tubes were regenerated through successive cleaning processes of sandblasting; cleaning with compress air, dipping in alcohol, cleaning with high pressure de-ionized water and in the end baking them in oven for 1 hour at 150^{0}C. The ceramic gap insulations of the tubes were checked after every cleaning process with a 5KV Megger in air. After cleaning, the insulation gaps showed marked improvement up to few tens of Giga ohms. The results obtained are shown in figure 2(a). The three tubes were tested for any vacuum leak and subsequently assembled back with their electrodes.

Ion source: Since the cathode electrode experienced breakdown in holding voltages, any spurious electrical contact with other electrodes had to be fixed. The ion source components get dirty when it runs for long durations. Often, it demanded for removal of the discharge paths caused by cesium contaminated surfaces. Thus, ion source was cleaned thoroughly. All the components were assembled using alignment kit with utmost care to avoid any spurious electrical contacts. It was baked and conditioned.

Cleaning of aluminium coated wall, roof of high voltage room, two high voltage decks: To avoid any discharge paths formed due to dirt, the aluminium coated wall, roof of the high voltage room, the two high voltage decks were thoroughly cleaned.

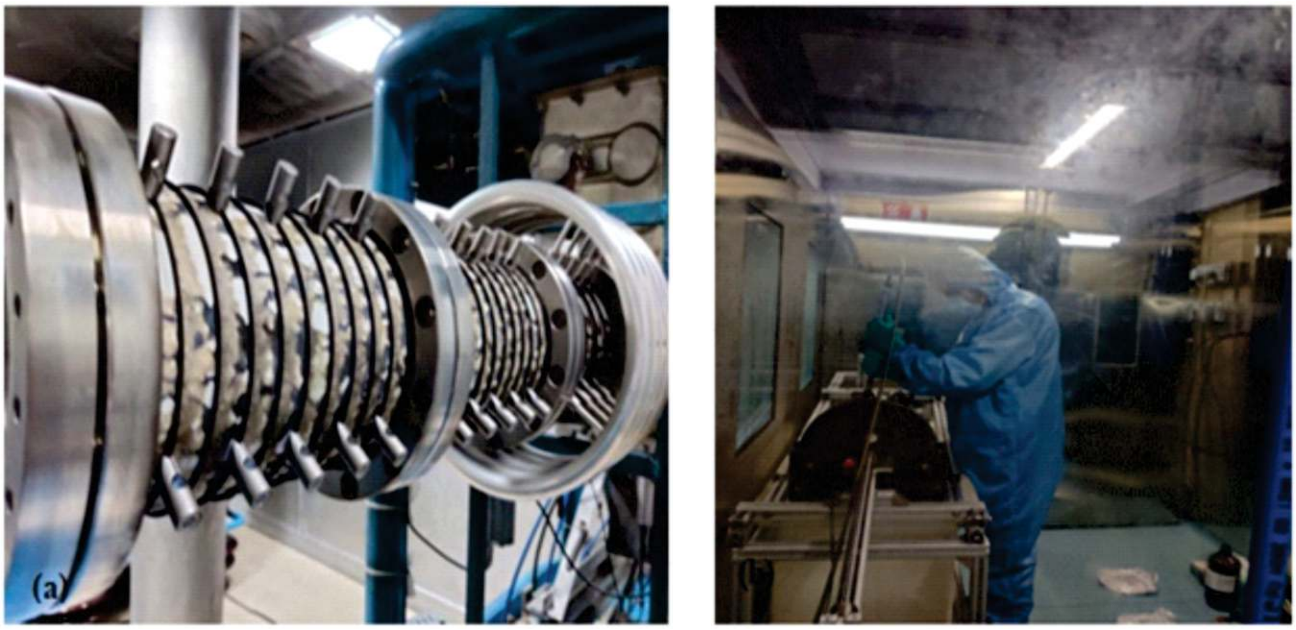
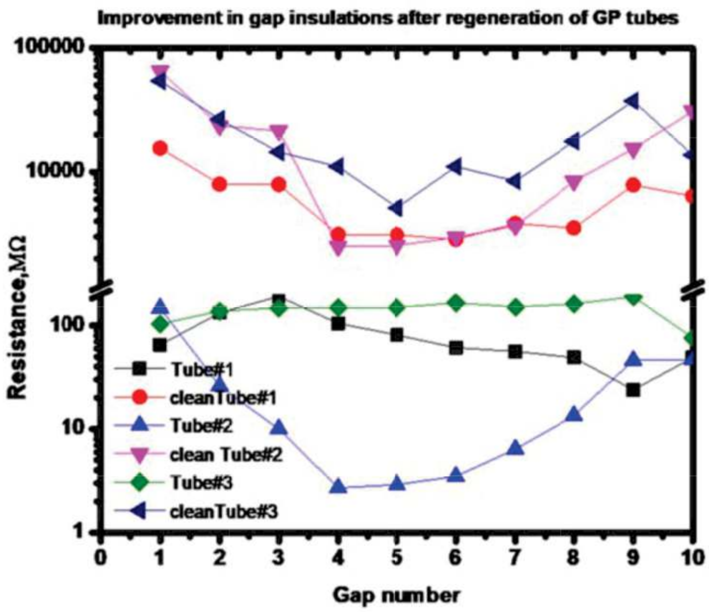


Figure1. (a) General Purpose (GP) Accelerating tubes with electrically shorted insulation gaps and (b) Cleaning of GP tubes using high pressure de-ionized water.



(a) (b)

Figure 2: (a) Observed improvement in gap insulations after cleaning (b) the damaged coolant (LOBS) in solid form inside the reservoir.

Cockcroft Walton High Voltage multiplier:The toroid head of Cockcroft Walton voltage multiplier was dismantled. All the components of the voltage multiplier stack were thoroughly cleaned with alcohol, dried with compressed air and re-assembled. The normal operation and stability of the accelerator system with its highest acceleration voltage, 200KV was achieved after conditioning.

Ion extraction system: The einzel lens assembly was thoroughly cleaned.

Grounding: The grounding of every power supplies of ion source components, two high voltage decks, isolation transformer and the Aluminum coated wall were checked.

Re-installation of injector beam line and conditioning

The injector beam line was reinstalled after regenerating its General Purpose accelerating tubes, ion source, extraction system and focusing lens. The re-installed beam line was checked to ensure that there was no vacuum leak. The beam line was pumped for creating vacuum and baked for 2 days. The resistors and toroids were mounted on the accelerating tubes after rechecking vacuum leak in the installed beam line.

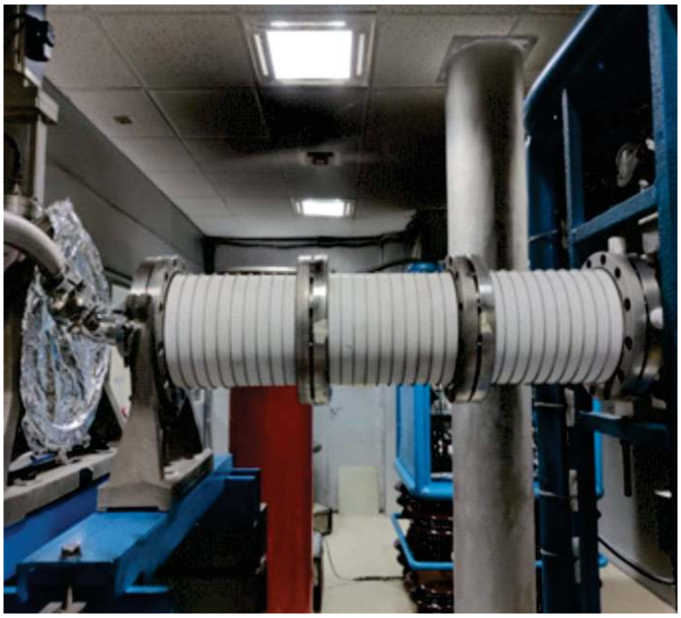


Figure 3. (a) Cleaning of Cockcroft Walton High Voltage multiplier, (b) Re-installed injector beam line.

The accelerator had been put on conditioning and testing for almost one week. Every component of the accelerator, from the ion source to the target station, was checked for their normal operation. However, with coolant on, the deck and cathode could hold very less voltages and showed fluctuations.

Ion source coolant: The coolant was found contaminated and we observed formation of solid lumps inside the coolant reservoir. It lost its insulation property and possibly forming a conducting path to the cathode voltage as well as the high voltage deck. The contaminated coolant was replaced with fresh coolant.

Beam Steering: Ion beams were generated in the ion source and transported up to the beam dump (Faraday Cup) after the target chamber. We observed steering of the beam towards –X side after the electrostatic quadrupole triplets (EQT) in the injector beam line, namely EQT-01. It was corrected by adjusting the entry point in +X direction.

Ionizer breakdown and replacement: The ionizer in the ion source broke down on 23 December, 2021 during an experiment. This ionizer lasted for around two years of routine operation. The ionizer was replaced, baked, and the beam was delivered within couple of days.

1.6.3 Development Activities

New Beam Development: Routinely attempts are being made to develop new ion beams in addition to improving ion intensities of various ion species. This academic year too, we could deliver a new beam, ^{130}Te for ion implantation experiments.

Automation of shutting down of the entire implanter accelerator: A source code was written and installed in the control system. The incorporated programme code was successful in shutting down the entire accelerator system except the high voltage. Soon we will be incorporating the high voltage part as well in the program.