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Citation: [AIP Conference Proceedings](#) **1640**, 129 (2015); doi: 10.1063/1.4905410

View online: <http://dx.doi.org/10.1063/1.4905410>

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# First Multicharged Ion Irradiation Results from the CUEBIT Facility at Clemson University

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**Abstract.** A new electron beam ion trap (EBIT) based ion source and beamline were recently commissioned at Clemson University to produce decelerated beams of multi- to highly-charged ions for surface and materials physics research. This user facility is the first installation of a DREEBIT-designed superconducting trap and ion source (EBIS-SC) in the U.S. and includes custom-designed target preparation and irradiation setups. An overview of the source, beamline, and other facilities as well as results from first measurements on irradiated targets are discussed here. Results include extracted charge state distributions and first data on a series of irradiated metal-oxide-semiconductor (MOS) device targets. For the MOS devices, we show that voltage-dependent capacitance can serve as a record of the electronic component of ion stopping power for an irradiated, encapsulated oxide target.

**Keywords:** highly charged ions, dielectric films, metal-oxide-semiconductor devices

**PACS:** 07.77.Ka, 68.49.Sf, 77.55.df

## INTRODUCTION

Electron beam ion traps and sources are devices which can be used to produce confined distributions of highly charged ions (HCIs) for atomic and materials physics research as well as research into plasmas with astrophysical and fusion-related relevance [1–3]. Here we provide a preliminary overview of a new laboratory that has been designed primarily for materials irradiation studies using a hybrid ion source from DREEBIT, GmbH<sup>1</sup>[4–11]. In the sections that follow we discuss the ion source and beamline and show first data obtained for an irradiated oxide system which was measured using a capacitance-voltage technique.

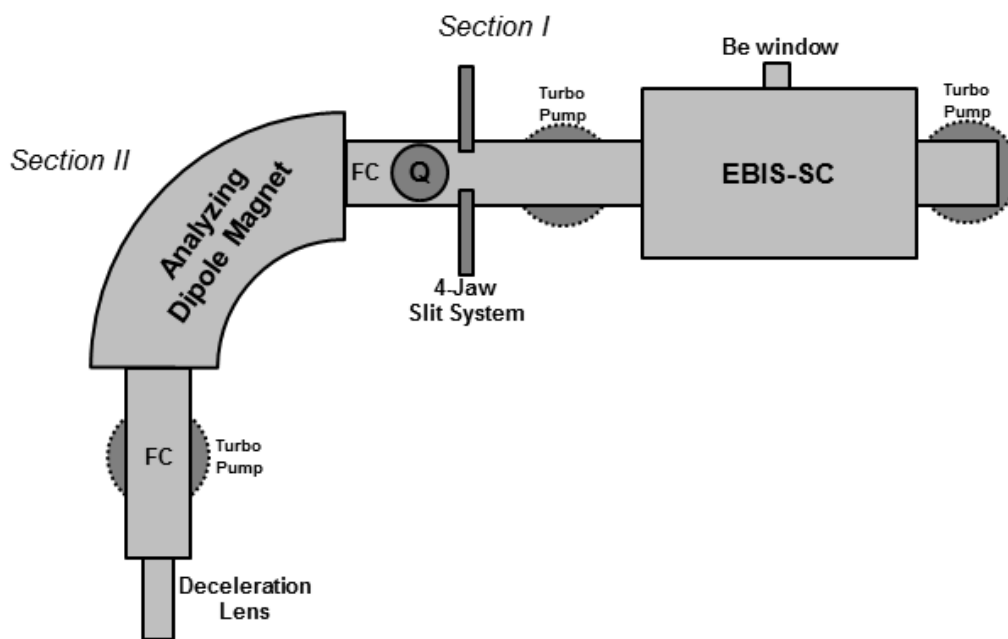
## EBIT LABORATORY

The Clemson University Electron Beam Ion Trap (CUEBIT) facility was funded by the National Science Foundation to establish a user-accessible laboratory that would focus on delivering decelerated beams of HCIs for target irradiation studies. The use of an EBIT or electron beam ion source (EBIS) for beam-based irradiation has a decades-long history; however, these facilities often piggyback onto atomic physics focused laboratories and, as such, have limited beam time and often include inflexible target preparation and mounting. The goal of CUEBIT was to provide HCI beams from an EBIT-based source with the ability to decelerate those beams and deliver them to flexible, and optimally user-defined target irradiation chambers or configurations.

Construction of the CUEBIT facility was initiated in 2010 and the ion source and beamline installations were completed in 2013. The facility is located in the Kinard Laboratory of Physics on the main campus of Clemson University and houses the ion source and beamline, which are described below. Additionally, the laboratory includes a thin film evaporation source and multiple target irradiation chambers which can be reconfigured as needed.

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<sup>1</sup> DREEBIT, GmbH, Dresden, Germany URL: [www.dreebit.com](http://www.dreebit.com)



**FIGURE 1.** A schematic layout of the ion source (EBIS-SC) and beamline (Sections I and II) in the CUEBIT facility. The Faraday cups and quadrupole deflector are labeled FC and Q, respectively. Detailed internal schematics for the EBIS-SC can be found in Ref. 12.

## Ion Source

The ion source at CUEBIT is an EBIS-SC machine which is qualitatively similar to the one described in Ref. 12. The electron beam is formed from a cathode emitter and directed through a three-section drift tube where it can ionize an injected species to form HCIs. Beyond the drift tube lies an extraction region where ions and electrons are separated. From the extraction region, ions leave the source and enter the beamline while the electrons are dumped into a collector. Both the collector and cathode are cooled with a closed cycle deionized water loop which cycles through an external laboratory chiller unit (ArcticChill P-Series)<sup>2</sup>. Surrounding the drift tube region is a 6 T closed-cycle-cooled superconducting magnet which provides compression for the central electron beam.

As in all EBIT/EBIS machines, bias voltages applied in the drift tube region along with the electron beam serve to confine ionized species axially and radially. Confinement times can be varied within the EBIS-SC by modulating the voltage of the drift tube section adjacent to the extraction region between “open” and “closed” trap configurations. Typical modes involve setting the drift tube at a low voltage such that ions with sufficient kinetic energy can surmount the axial potential continuously (leaky mode) or pulsing the drift tube voltage to lower the axial potential over fixed time intervals (pulsed mode). The central drift tube, which is 20 cm in length and has a rated upper voltage of 20 kV, has currently been run up to 15 kV and has been used to create HCIs from injected Ar gas up to Ar<sup>13+</sup>. Future design configurations will include injection of desired species through volatile compounds placed in-line with the gas injection port of the EBIS-SC as well as ion-based injection through a quadrupole beam deflector mounted in the beamline.

The charge state configuration of ions within the trap region of the EBIS-SC can be probed through either the extracted ion beam (see below) or through the measurement of x-ray spectra. A spectroscopy port for x-rays is mounted on the EBIS-SC vessel perpendicular to the electron beam axis direction with a line of sight to the central drift tube. The drift tube, cathode, and collector are at the center of the EBIS-SC vacuum vessel and are oil-free pumped using a turbomolecular pump. This region is separated by a Be window from an outer evacuated volume which houses

<sup>2</sup> ArcticChill USA, Newberry, SC URL: [www.arcticchillergroup.com](http://www.arcticchillergroup.com)

the superconducting magnet and is also oil-free pumped with a turbomolecular pump. The spectroscopy port, which includes a second Be window, lies on the exterior of this magnet volume. The current base pressures for both the drift tube and magnet-containing volumes are in the  $10^{-10}$  mbar range. A third vacuum region is accessible behind a linear translator mounted on the cathode which is installed to facilitate the removal and replacement of the cathode while maintaining vacuum elsewhere within the machine. The cathode, extractor, and drift tube current/voltage settings, superconducting magnet operation, and vacuum pump operations are all interfaced to be operated from a LabView front-end software package written by DREEBIT, GmbH.

## Ion Beamline

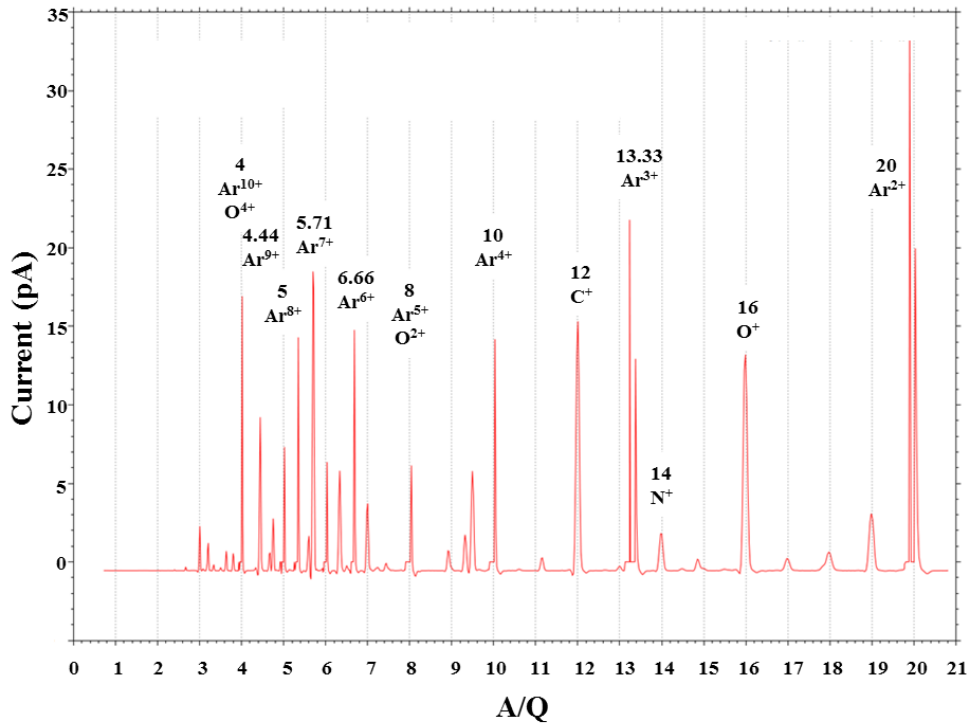
Connected to the EBIS-SC ion source are two additional vacuum sections (Sections I and II) which constitute the beamline for the CUEBIT facility as shown in Fig. 1. Both sections are oil-free pumped with turbomolecular pumps and are separated from the ion source and each other by pneumatically-controlled gate valves. Within Section I, immediately adjacent to the ion source, are mounted an Einzel lens and deflector assembly. Both are used to focus and deflect ions extracted from the source into a retractable Faraday cup mounted within this section. Also mounted in Section I are a quadrupole beam deflector and a 4-jaw slit system. The quadrupole deflector is currently unused; however, it can be employed as an injection point to transport ions back into the central drift tube or trap region for further ionization. This has been demonstrated in other setups, see *e.g.* Ref. 13, to facilitate the ionization of species that are not easily injected into the source in either gaseous or volatile form. The 4-jaw slit is mounted at the focus point of the analyzing dipole magnet of Section II and serves as an aperture for the beam passing out of Section I and can be used to reduce the beam diameter or to deduce its size and position.

Extracted HCI beams focused through Section I are brought into Section II through an analyzing dipole magnet. A second deflector assembly lies at the exit of the magnet and is used to bring the beam into alignment for this section's Faraday cup or into user-defined detector assemblies mounted in the target region downstream. The analyzing dipole magnet has a deflection angle of  $90^\circ$ , a bending radius of 350 mm, and produces a maximum field of 0.4 T at a 91.2 A induction current. A water cooling loop, also powered by the laboratory chiller, serves to cool the magnet coils, and a Hall probe is mounted within the magnet yokes and interfaced to the laboratory computer to provide field strength readings. The Section II Faraday cup is mounted at the analyzing magnet's exiting focus point, and the collection of current readings under varying field strengths in this cup can be used to generate a spectrum of the ion species extracted from the EBIS-SC source. An example spectrum (Fig. 2), one of the first obtained during on-site commissioning of the facility, shows Ar charge states up to  $\text{Ar}^{10+}$  as well as ions of C, N, and O formed from background contamination.

As with the ion source, the controls for the lenses, deflectors, Faraday cups, 4-jaw slits, beam deflector, and analyzing magnet are interfaced and controlled from the laboratory's front-end software. This also includes the two vacuum systems (gate valves and pumps) which are interlocked so that they can self-isolate should the vacuum pressure in adjoining sections rise beyond predetermined setpoints. Base pressures in Sections I and II are currently in the  $10^{-10}$  mbar range.

## Target Region

The target region of the CUEBIT facility refers to the delivery point for HCI beams extracted and focused from the EBIS-SC ion source. Extending into this region is a custom-designed deceleration lens that is configured to provide up to 100X reduction in the delivered beam energy. The lens has an inner diameter of 40 mm and is constructed upon a DN100CF (6 in. CF) flange. Given the flexible operating procedures designated for CUEBIT as a user facility, this base flange is considered the standard mounting point for any desired chamber or experimental end point configuration. To date we have connected both DN100CF and DN160CF (6 in. CF and 8 in. CF) six-way crosses as target chambers in this region. For the first data described in the next section, a DN100CF cross, which was oil-free turbo pumped with a translator and home-built Faraday cup were used. The system was configured to be load-lockable through a gate valve and magnetic linear translator and samples were mounted on Omicron-style platens that could be placed alongside the Faraday cup and translated into the decelerated HCI beam focus position.



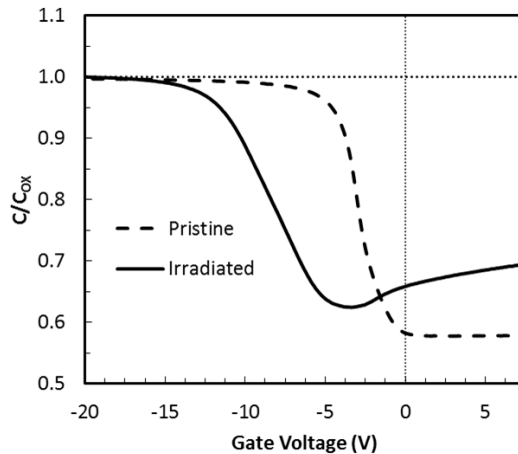
**FIGURE 2.** An extracted ion spectrum obtained in the Section II Faraday cup as the analyzing dipole magnet field strength was varied. Measured current results are plotted versus the mass-to-charge ratio ( $A/Q$ ) that corresponds to the analyzing magnet setting. These data were taken under Ar gas injection into the ion source and the accelerating voltage of the source was set to give an energy per charge of 4.5 keV/Q.

## EXPERIMENT

As a first experiment using extracted ions at CUEBIT, unfinished metal-oxide-semiconductor (MOS) devices were introduced into the target region and irradiated with  $\text{Ar}^{Q+}$  ions. The goal was to explore whether charge-state-dependent stopping effects could be resolved in capacitance-voltage (C-V) measurements. A wafer (3-in. p-type Si(100)) purchased from Silica-Source, Inc. with a resistivity in the 1-10 ohm-cm range was the starting material for these devices. The wafer was precleaned prior to oxide growth using a standard RCA clean (1:1:5 solution of  $\text{NH}_4\text{OH} + \text{H}_2\text{O}_2 + \text{H}_2\text{O}$ ), etched with dilute 1% HF to remove any native oxide, and triple rinsed in deionized water. An oxide film was then grown on the wafer in an oxidation furnace under steam flow to a nominal thickness of 1750 Å ( $1746 \text{ Å} \pm 41 \text{ Å}$ ). A backside Ohmic contact was prepared on the wafer by HF etching the backside, depositing 0.5  $\mu\text{m}$  of Al from a thermal evaporator onto the cleaned surface, and then sintering the wafer at 450° C for 30 mins. in a nitrogen environment. The prepared, oxidized wafer with Al backside contact was then diced into multiple square samples (~12 mm) to accommodate the Omicron-style sample mount of our ion irradiation setup.

For the irradiations, the samples were load-locked into the target region and exposed to focused beams of  $\text{Ar}^{Q+}$  ions ( $Q=1, 4, 8, \text{ and } 11$ ) that were all decelerated to have kinetic energies of approximately 1 keV. For the charge states  $Q=4, 8, \text{ and } 11$ , multiple samples were exposed at different total ion doses in the range of  $5 \times 10^{11} - 5 \times 10^{12}$  ions/cm<sup>2</sup>. The base pressure within the target region during exposures was in the  $10^{-8}$  mbar range. Following each irradiation, the exposed sample was removed and transported to the thermal evaporator so that ~ 1 mm diameter top Al contacts could be deposited to form multiple, individual MOS capacitors across the sample surface. An example is shown in Fig. 4(a) where 25 devices have been created on one sample.

Each individual MOS device on a given sample was characterized using a C-V measurement. A typical high frequency C-V signature for an unirradiated MOS capacitor is shown as the dashed line in Fig. 3. Under applied gate voltage, the C-V curve shows accumulation behavior at negative voltages where the measured capacitance is due to the intrinsic capacitance of the MOS oxide layer. At positive gate voltages the measured value drops due to the



**FIGURE 3.** Two high frequency C-V curves taken on pristine (dashed line) and irradiated (solid line) MOS devices (Al/SiO<sub>2</sub>/Si) with an  $\sim 1750$  Å oxide layer. The irradiated device was constructed on a sample exposed to 1 keV Ar<sup>4+</sup> ions at a dose of  $4.58 \times 10^{11}$  ions · cm<sup>-2</sup>.

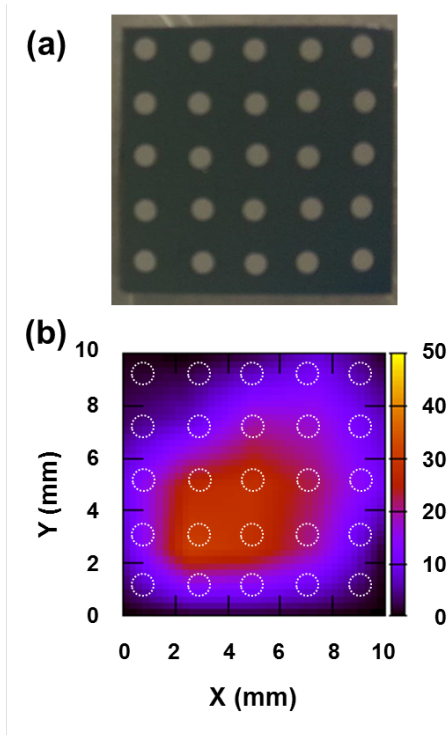
depletion layer capacitance. Between these extremes, the curve drops at a point in voltage that is near to the so-called flatband voltage ( $V_{FB}$ ). This is the point where, for an ideal system, the gate voltage equals the work function difference between the Al gate and the Si substrate ( $\approx 0.8$  eV)[14]. In reality, for our grown oxides, the measured pristine  $V_{FB}$  values are  $\sim 3.8$  V.

More generally,  $V_{FB}$  serves as a measure of the detailed conditions of the oxide and its interfaces. For the preliminary results discussed here, this applies to additional charges, such as excited holes, left by the ion irradiation step. For example, Fig. 3 also shows C-V data obtained for a device irradiated by 1 keV Ar<sup>4+</sup> ions (solid line). Here there is a clear shift in the  $V_{FB}$  position toward more negative gate voltage values. This is consistent with radiation damage seen in other systems irradiated by gamma rays and UV radiation[15, 16]. Therefore, it is through the shifted  $V_{FB}$  values, taken relative to the pristine value measured for our prepared wafer, that we track the energetics of energy dissipation for our Ar<sup>Q+</sup> ions that have impacted the MOS oxide layer.

## PRELIMINARY RESULTS

Given the focused nature of the HCI beams extracted and directed into the target region, it has been observed that there is a distribution of dosed regions across each of the samples irradiated for this measurement. This can be seen in Fig. 4(b), where the measured  $V_{FB}$  data taken across multiple deposited Al gates (gate positions indicated by the white overlaid circles) has been interpolated across the entire sample surface. The figure clearly represents the current density profile of the irradiating beam in terms of the induced shifts it generates in the  $V_{FB}$  values for the subsequently deposited MOS devices. This result, which is seen for all the charge states explored, indicates that the dose of ions striking the SiO<sub>2</sub> is recorded by the C-V extracted  $V_{FB}$  shifts. Such a dose dependence is consistent with previous results for HCIs and devices, where each ion impact is determined to produce a measurable change in the irradiated target material[17].

In addition, the results obtained to date for multiple charge states indicate that there is a significant enhancement in the induced  $V_{FB}$  shift as the charge state of the incoming beam is increased. This can be quantified by measuring the  $V_{FB}$  shift across multiple ion doses for fixed incident charge states so that a normalized value of the  $V_{FB}$  shift induced per incident ion can be obtained. Although preliminary, the normalized results show an enhancement in the normalized shift per ion, which grows monotonically across our charge state data, from  $1.14 \times 10^{-12}$  V/ion for Ar<sup>1+</sup> ions to  $1.12 \times 10^{-11}$  V/ion for Ar<sup>11+</sup> ions. We note that this approximately order of magnitude increase occurs for ions with a fixed incident kinetic energy of 1 keV. Therefore, we can interpret this result as a reflection of the differences in potential energy for the two charge states (15 eV for Ar<sup>1+</sup> and 2004 eV for Ar<sup>11+</sup>). In simplest terms, the sensitivity of the C-V results to subsurface oxide damage has served as a record of the enhanced potential energy dissipation of



**FIGURE 4.** (a) Deposited Al dots on an  $\text{Ar}^{4+}$  irradiated  $\text{SiO}_2/\text{Si}$  wafer with an  $\sim 1750 \text{ \AA}$  oxide layer. (b) Interpolated image of the measured  $V_{FB}$  shifts obtained for each MOS device from high frequency C-V spectra. Units for color scale are volts and the overlaid white circles correspond to device positions from (a).

the higher charge state ions as they impact on and are implanted into the oxide layer.

## SUMMARY

A new HCI source and beamline have been constructed and installed at Clemson University as a user facility for materials irradiation research. This EBIS-SC based facility has been used to create beams of HCIs from introduced Ar gas and has the necessary installed features to accommodate introduction of other species through volatile- or ion-based injection into the source. Preliminary data on irradiated  $\text{SiO}_2$  encapsulated into MOS devices have been interpreted through C-V measurements. These results appear to confirm that dose- and charge-dependent effects can be recorded for HCI irradiation on oxides using this method.

## ACKNOWLEDGMENTS

The authors acknowledge the support of the National Science Foundation (NSF-DMR-0960100), DARPA (ARO Grant No. W911NF-13-1-0042), and the Clemson University College of Engineering and Science.

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