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MIAMI: Microscope and ion accelerator for materials investigations

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A transmission electron microscope (TEM) with *in situ* ion irradiation has been built at the University of Salford, U.K. The system consists of a Colutron G-2 ion source connected to a JEOL JEM-2000FX TEM via an in-house designed and constructed ion beam transport system. The ion source can deliver ion energies from 0.5 to 10 keV for singly charged ions and can be floated up to 100 kV to allow acceleration to higher energies. Ion species from H to Xe can be produced for the full range of energies allowing the investigation of implantation with light ions such as helium as well as the effects of displacing irradiation with heavy inert or self-ions. The ability to implant light ions at energies low enough such that they come to rest within the thickness of a TEM sample and to also irradiate with heavier species at energies sufficient to cause large numbers of atomic displacements makes this facility ideally suited to the study of materials for use in nuclear environments. TEM allows the internal microstructure of a sample to be imaged at the nanoscale. By irradiating *in situ* it is possible to observe the dynamic evolution of radiation damage which can occur during irradiation as a result of competing processes within the system being studied. Furthermore, experimental variables such as temperature can be controlled and maintained throughout both irradiation and observation. This combination of capabilities enables an understanding of the underlying atomistic processes to be gained and thus gives invaluable insights into the fundamental physics governing the response of materials to irradiation. Details of the design and specifications of the MIAMI facility are given along with examples of initial experimental results in silicon and silicon carbide. © *2011 American Vacuum Society.* [DOI: 10.1116/1.3543707]

I. INTRODUCTION

Transmission electron microscopy (TEM) with *in situ* irradiation allows the internal microstructure of materials to be investigated at the nanoscale while they are being irradiated, enabling dynamic microstructural effects to be observed. During irradiation competing processes cause radiation damage to evolve and by being able to explore this development as it occurs rather than merely observing the end states accessible in *ex situ* studies, insights into the underlying atomistic processes can be achieved and fundamental understanding into the response of materials to radiation damage can be gained.

Transmission electron microscopy with *in situ* ion irradiation was first performed ca. 1961 by Pashley and Presland using O^- ions emitted from the tungsten filament of a TEM at Tube Investment Laboratories, Cambridge, U.K.[1] The first system to interface a dedicated ion beam system with a TEM was developed around 1968 by Nelson and co-workers at the Atomic Energy Research Establishment, Harwell, U.K.[2–4] Since then, over 30 facilities have been constructed in Japan, France, USA, Canada, and China. A recent review of these facilities detailing their history and design can be found in Ref. 5.

Microscope and Ion Accelerator for Materials Investigations (MIAMI) is a new facility which has been established in the U.K. It is capable of delivering ionic species from H to Xe with energies from 500 eV to 100 keV for singly charged ions. This range allows experiments with light ions, such as He, at energies low enough for them to come to rest in the thickness of a TEM sample (typically 50–100 nm) or heavier ions at higher energies which can create large numbers of displacements per atom (DPA). The fluxes achievable allow, for example, materials to be irradiated to fluences corresponding to the He concentrations or DPA commensurate with those experienced by components in fission and fusion reactors at the end of their expected lifetimes. Thus, the responses of materials can be explored to these very high levels of damage leading to an understanding of the underlying mechanisms, the ability to predict their behavior and to develop improved materials for these applications.

This new facility distinguishes itself from other TEMs with *in situ* ion irradiation currently in operation around the world by being able to deliver ions at both low and high energies, as discussed above, and of almost any species by virtue of its ability to run from either gaseous or solid state feed material.

II. OVERALL DESIGN

The MIAMI facility features an ion source which can be floated up to 100 kV with the additional ion acceleration performed after a bending magnet—the high-voltage section and the earthed cage which when surrounds it can be withdrawn from the TEM to allow maintenance on the beamline and deflection systems. The overall layout is shown in Figs. 1 and 2. After passing through the bending magnet and

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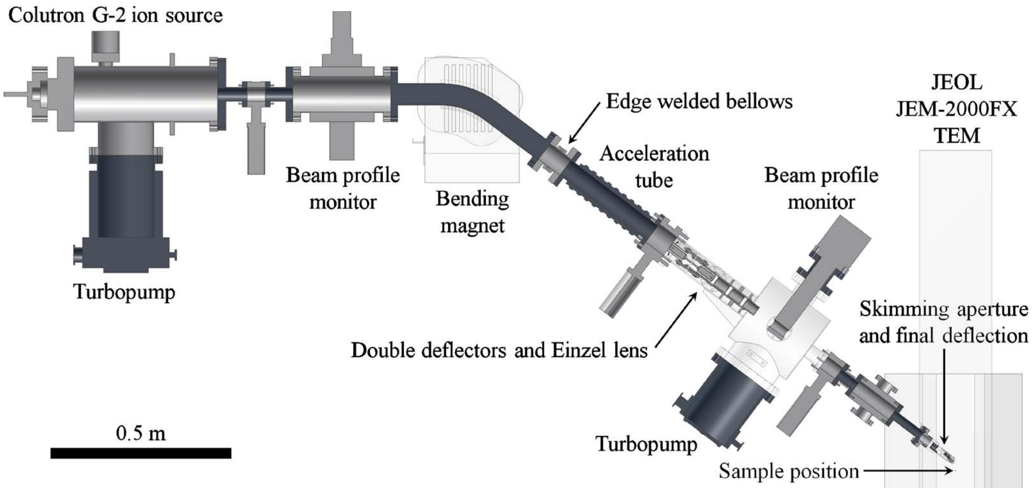

FIG. 1. (Color online) Layout of ion in the beam production and transport systems.

through the acceleration tube, the beam is manipulated using a double set of electrostatic deflectors and an Einzel lens before entering the TEM column via a port originally designed for a high take-off angle x-ray detector. Once inside the TEM column the ion beam passes through an aperture in a beam-skimming diaphragm which is used for beam alignment and continuous flux measurement during sample irradiation before being electrostatically deflected onto the sample position.

III. ION SOURCE

A Colutron G-2 ion source (shown in Fig. 3) has been installed as this can produce high beam currents of light ions such as helium and operate with either a gas or a solid charge.[6,7] Energies from 0.5 to 10 keV for singly charged ions can be produced by the source itself with postacceleration performed as described below in Sec. IV B. The unit also features an Einzel lens, vertical deflectors, Wien filter, and beam shape adjustment.

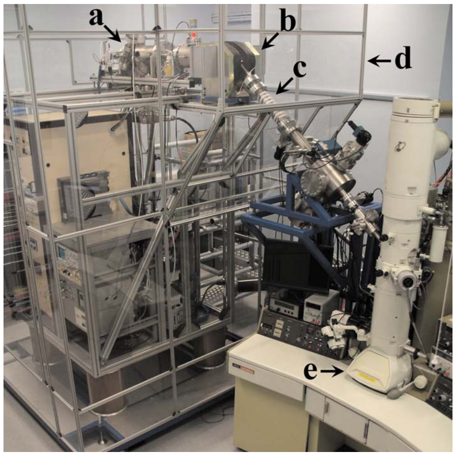

FIG. 2. (Color online) MIAMI facility (TEM with *in situ* ion irradiation): (a) Colutron G-2 ion source (see Sec. III); (b) bending magnet (see Sec. IV); (c) acceleration tube (see Sec. IV); (d) earthed frame to allow the equipment contained within to be safely floated up to 100 kV; and (e) JEOL JEM-2000FX TEM (see Sec. VI).

IV. BEAM TRANSPORT

A. Bending magnet

After leaving the ion source the beam passes through a bending magnet which deflects the ions through an angle of 53° to bring them on to the axis of the high take-off angle x-ray port of the TEM and also acts as a mass-energy selection device—see Fig. 4. By placing the magnet before the postacceleration stage the field strength required to deflect ions is greatly reduced when operating at higher energies.

B. Acceleration tube

Once the beam has passed through the magnet the ions can be accelerated to higher energy by an in-house designed nine stage acceleration lens and resistor chain which spans the gap between the high-voltage and earthed sections—see Fig. 5. This is based on design implemented previously at the

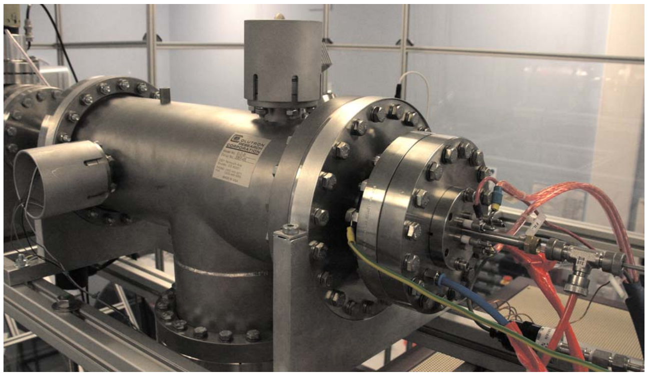

FIG. 3. (Color online) Colutron G-2 ion source used to produce ionic species from either gas or solid state charges with energies of 0.5–10 keV for single charged ions.

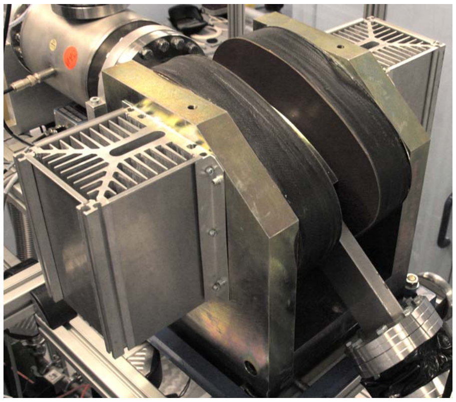

FIG. 4. (Color online) Bending magnet used to deflect ion beam through 53° from horizontal.

University of Salford.[8] By measuring the current down the resistor chain the voltage to which the high-voltage section is being floated and across which the ions are being accelerated can be monitored.

C. Einzel lens and double deflectors

To provide beam positioning and additional focusing, an Einzel lens and set of double deflectors have been installed before the beamline chamber—see Fig. 6. The chamber is intended to provide the possibility of irradiating either TEM samples or bulk material *ex situ* to the TEM. By using double deflectors the beam can be shifted and tilted as desired allowing rastering across a sample. However, for the purposes of normal alignment it is sufficient to utilize only one set of deflectors as at the distance involved the angular deflection to achieve the required shifts is small.

D. Final deflection

The high take-off angle port of the TEM does not provide adequate line of sight to the sample position and so an electrostatic deflection system has been designed in-house which is inserted inside the port itself—see Fig. 8. Bent plates with voltages of equal magnitude and opposite polarity are used so the ion beam follows a line of close-to-zero potential so as to reduce focusing effects.

FIG. 5. (Color online) Nine stage acceleration tube used to postaccelerate ions after the bending magnet (Ref. 8).

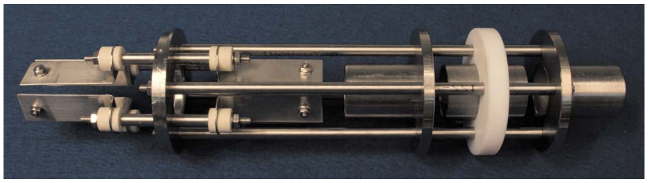

FIG. 6. (Color online) Double deflectors (left) and Einzel lens (right) allow x/y tilt/shift and focusing along the beamline between the acceleration tube and the microscope.

It is desirable to have the smallest angle possible between the ion and electron beams in order to have access to the widest range of sample tilts, so as to be observing in a direction as close as possible to the direction of irradiation and to avoid high degrees of sample tilt which limit the useable thin area in a sample. Due to the space constrictions of the port diameter (20 mm) and the electric field strength required to deflect ions which have been accelerated across 100 kV, the minimum angle achievable between the ion and electron beams is 25°—this compares well with other instruments being bettered by only 4 out of 28 of the other TEMs interfaced to ion beam systems which have been constructed around the world.[5]

An aperture is placed at the exit from the final deflection system to shield the electron beam of the TEM from the electric field and insulating surfaces. In order to test for interference with the imaging capabilities of the TEM caused by the electrostatic field, lattice images have been observed while the voltage applied to the deflection plates has been increased from zero to its maximum value: no degradation of the image was observed.

V. BEAM MONITORING AND DOSIMETRY

A. Beam profile monitoring

Two National Electrostatic Corporation beam profile monitors have been installed on the beamline—one immediately after the ion source and another on the beamline chamber. The latter is designed so as to provide the option of retracting it from the beamline to make room for irradiation of samples in the chamber as described above in Sec. IV C. In both profilers a helical wire is rotated such that it sweeps through the ion beam generating secondary electrons which are then collected as a current that is amplified and displayed on an oscilloscope giving the beam profile in two orthogonal directions.

B. Skimming diaphragm

In order to align the ion beam such that it enters the final deflection on the zero potential line between the deflection plates, a skimming diaphragm is placed just before the final deflection system—see Fig. 7. This has the dual function of also providing a measurement of the ion beam current striking the diaphragm during experiments. Due to the space constraints within the TEM it is not practical to continuously measure ion beam current after the final deflection stage (Fig. 8).

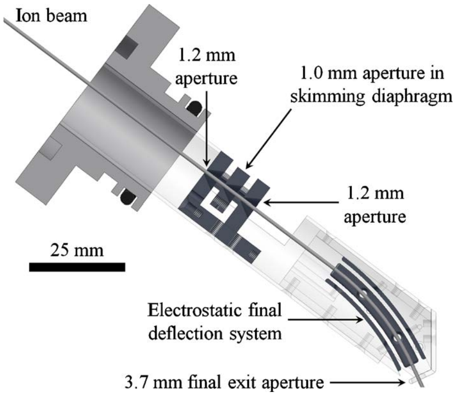

FIG. 7. (Color online) Diagram showing skimming diaphragm and final deflection system—the 1.0 mm skimming diaphragm defines the diameter of the ion beam and along with proper alignment ensures that it enters the final deflection system on the equipotential line.

C. Current metering rod

Ion beam current at the sample position can be measured using an in-house designed and constructed current metering device which enters the TEM in the same manner as a TEM sample rod—see Fig. 9. An entrance aperture is located so as to be exactly at the TEM sample position and the transmitted ion beam current is measured on a collector plate. An intermediate aperture of larger diameter than the entrance aperture is maitained at a voltage of −30 V to suppress secondary electrons.

In order to ensure that the entrance aperture is centered on exactly the correct position, the electron beam of the TEM is detected on the current metering rod which is then centered using the *x* and *y* specimen shift controls of the microscope.

By calibrating the ion beam current measured on the skimming diaphragm to that on the current metering rod, the latter can be used to detect and record any changes in ion beam flux during an experiment.

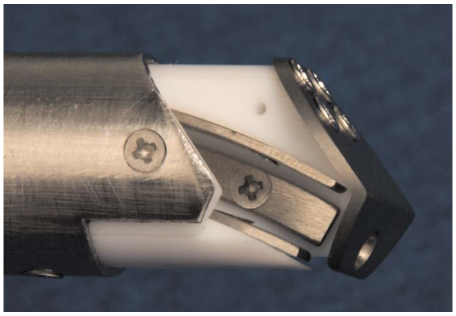

FIG. 8. (Color online) Close-up of final deflection system with internal components partially extracted to show one horizontal and both vertical electrostatic deflection plates. The end plate is designed to shield the electron beam of the TEM from the insulating materials and electrostatic field of the final deflection system; it is made of tungsten with an aperture significantly larger than the diameter of the ion beam to reduce sputtering onto the sample and other surfaces.

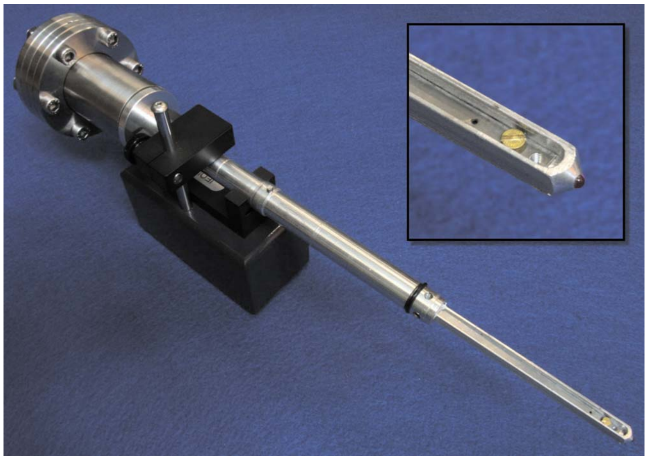

FIG. 9. (Color online) Current metering rod which can be inserted into the TEM in order to measure the ion beam flux at the sample position. Secondary electron suppression is performed by a −30 V potential applied to a diaphragm positioned between the entrance aperture and collection plate. The inset shows close-up of entrance aperture for ion beam and positioning sapphire.

D. Retractable Faraday cup

A retractable Faraday cup has been designed and constructed in-house to allow the ion beam to be monitored after the final deflection stage and at height just above the sample so that the sample can remain its position during the measurement—see Fig. 10. This provides a check of both ion beam current and position calibrated against the current metering rod. The Faraday cup is mounted on an adapted cathodoluminescence detector stalk which provides both positioning and electrical feedthroughs.

E. Gatan Faraday cup

The Gatan liquid nitrogen cooling rod available at the facility is fitted with a miniature Faraday cup which can also be used for ion beam current measurements.

VI. TRANSMISSION ELECTRON MICROSCOPE

A. JEOL JEM-2000FX

A JEOL JEM-2000FX was selected as it features a high take-off angle x-ray (37° from horizontal) port allowing the

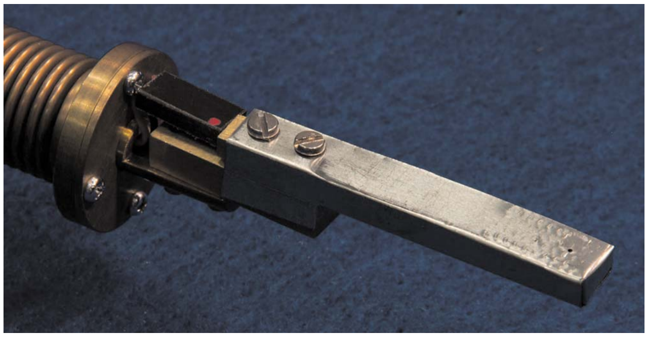

FIG. 10. (Color online) Retractable Faraday cup used to monitor ion beam current just above sample position during experiments.

TABLE I. Summary of MIAMI facility specifications.

Ion accelerating voltage	0.5–100 kV
Ion species (across full energy range)	H to Xe
Ion flux (depends on species and energy)	Typical for 10 keV He: 3×10^{13} ion cm^{-2} s^{-1}
Angle between ion and electron beams	25°
Electron beam accelerating voltage	80–200 kV
Magnification range	50–800 k
Resolution (lattice image)	0.14 nm
Resolution (point image)	0.31 nm
Sample tilt	Depends on sample holder, typically ±20°
Digital imaging and video capture	Gatan ES500W 1.4 megapixel and Gatan ORIUS SC200 4 megapixel
Sample temperature ranges	100–373 K or RT to 1273 K

ion beam to be introduced at a relatively small angle with respect to the electron beam. Unfortunately this port does not provide direct line of sight to the sample position and hence the necessity to employ electrostatic deflection within the column to make the ion beam incident upon the sample. However, this does mean that the angle between the ion and electron beams is reduced from 53° along the axis of the port to 25° at the point at which the two beams and sample all intersect. The maximum electron acceleration voltage is 200 kV and the resolution of the instrument allows lattice imaging to be performed on suitable samples—see Table I for further details.

B. Sample holders

Single tilt, double tilt, heating double tilt, and cooling double tilt holders are all available at the facility. These give access to a wide range of sample orientations and sample temperatures from 100 to 373 K or RT to 1273 K. Further sample holders will be made available in the near future.

C. Digital imaging

Two digital imaging systems have been installed on the system: a Gatan wide angle ES500W 1.4 megapixel charge-coupled device (CCD) camera and a bottom-mount Gatan ORIUS SC200 4 megapixel CCD camera. This combination allows both lower magnification (for the monitoring of larger defect structures and/or areas at higher video frame rates) and higher magnification imaging to be performed, respectively, with easy switching between the two cameras. The ORIUS SC200 also allows digital imaging of diffraction patterns for qualitative and quantitative analysis of processes such as changes in crystallography or amorphization.

VII. VACUUM SYSTEM AND VIBRATION ISOLATION MEASURES

The ion beam system is evacuated by two turbomolecular pumps each backed by a dedicated rotary pump. The turbomolecular pumps are magnetically levitated to reduce vibration of the beamline which could degrade microscope performance. In addition, each backing line has a 40 l reservoir tank to allow the rotary pumps to be switched off for a period time (dependent on gas load) that is required for high resolution imaging in the TEM. However, this has not yet been found to be necessary. The standard sputter ion pump of the JEOL JEM-2000FX has been replaced with a magnetically levitated turbomolecular pump to improve the ability of the microscope vacuum system to handle additional gas loads from the implanter and, particularly, to assist in the removal of inert gases such as He.

In addition to the aforementioned measures to reduce vibrations affecting the TEM, edge-welded bellows have been

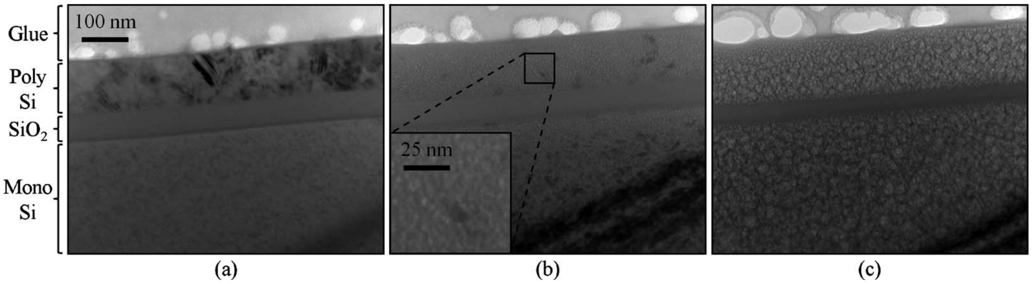

FIG. 11. Three fluence steps from a 6 keV He irradiation of monocrystalline and polycrystalline silicon at room temperature: (a) virgin sample; (b) after a fluence of 6.5×10^{16} ions cm^{-2}, small helium bubbles have appeared in both monocrystalline and polycrystalline materials, much of the diffraction contrast has been lost from the polycrystalline grains, and interstitial clusters can be seen in the monocrystalline material; and (c) after a fluence of 1.3×10^{17} ions cm^{-2}, the helium bubbles are much larger and significant swelling is visible (and measurable in the polycrystalline layer as an increase in its width). Concomitant shrinkage of the oxide layer is believed to be due to viscoelastic effects caused by the helium irradiation. TEM imaging conditions: bright field, off zone, and underfocus. The scale marker in panel (a) applies to the main images in all three panels.

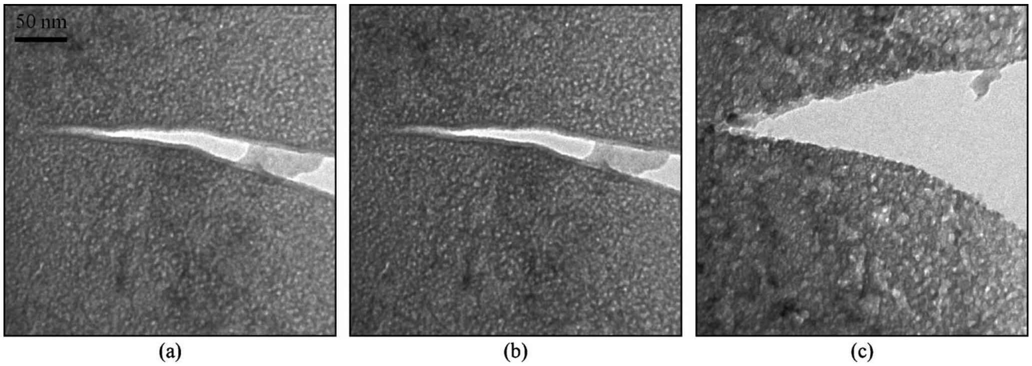

FIG. 12. Irradiation of helium bubbles in silicon carbide with 35 keV Ar at 900 °C. Panel (a) shows the helium bubbles at the beginning of the experiment and (b) shows the same area after 20 min at 900 °C—note that there is no change due to either the temperature or electron beam. Panel (c) shows the same area after a fluence of 10^{15} ions cm^{-2}. TEM imaging conditions: bright field, off zone, and underfocus. The scale marker applies to all three panels.

placed at either side of the beamline chamber. These also aid in the assembly and disassembly of the system during maintenance tasks.

VIII. FACILITY SPECIFICATIONS

A summary of the capabilities and specifications of the MIAMI facility is given in Table I.

IX. EXAMPLES OF EXPERIMENTAL RESULTS

A. 6 keV He irradiation of monocrystalline and polycrystalline silicon

In order to explore helium bubble nucleation and growth in monocrystalline and polycrystalline silicon, a trilayer consisting of monocrystalline and polycrystalline layers separated by a layer of oxide was irradiated with 6 keV He. Considerable levels of swelling were observed in both Si layers coupled with a reduction in the width of the oxide layer. Figure 11 shows three steps in this process. By performing this experiment *in situ* it was possible to continuously monitor this process and to directly compare the effects in the two types of silicon at identical fluence steps in exactly the same area of the sample with constant imaging conditions in the TEM.

B. 35 keV Ar irradiation of silicon carbide at 900 °C

Helium bubbles were created by 6 keV He irradiation of silicon carbide at 700 °C using the *in situ* facility. In a separate experiment, a further annealing stage was performed to 900 °C with no further change to bubble size and distribution. The sample was then irradiated with 35 keV Ar resulting in bubble growth and coalescence as shown in Fig. 12.

X. SUMMARY

A TEM with *in situ* ion irradiation, the MIAMI facility, has been constructed at the University of Salford, U.K. This new facility is one of only two in Europe and approximately 11 currently operating around the world. It is capable of delivering light ions such as helium at the low energies required for them to come to rest within the thickness of a TEM sample and also to produce heavier higher energy inert or self-ions in order to introduce large numbers of atomic displacements. The system has been designed, developed, and constructed in-house and is now being used to pursue investigations into a wide range of materials with applications in the nuclear, semiconductor, and nanotechnology industries.

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