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Transmission electron microscopy with in-situ ion irradiation: Facilities and community

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ABSTRACT

Whilst there is a clear scientific and technological need for the technical capabilities of transmission electron microscopes with in-situ ion irradiation, it also requires a collaborative community of international researchers to support such facilities in successfully meeting this demand. Instruments of this type serve to provide fundamental understanding of the mechanisms which drive changes in materials important to nuclear fission and fusion energy, the semiconductor industry, quantum information systems, space travel, astronomy, geology and many more applications. As these areas continue to evolve and the instrumentation possibilities expand, the capacity of in-situ ion irradiation facilities must also develop hand-in-hand with the user community to deliver an ever-greater diversity of high-fidelity extreme-environment experimentation. Future directions for the field, such as miniaturization from MEMS/microfluidic devices and advanced controls with ML-based analysis, continuously emerge to advance both the hardware and software which support the coupling of TEMs with ion beams. This review sets out to provide up-to-date insights into the community and advancement of current, and development of future, facilities which have the potential to further unlock access to the nanoscale exploration of coupled extreme environments crucial to many of the important science and engineering challenges we face today.

1. Introduction

Materials exposed to ionizing irradiation environments experience direct effects on the atomic scale leading to evolution of their structure on the nano- and microscales. This can then result in alteration of their properties with important implications for their ability to perform in

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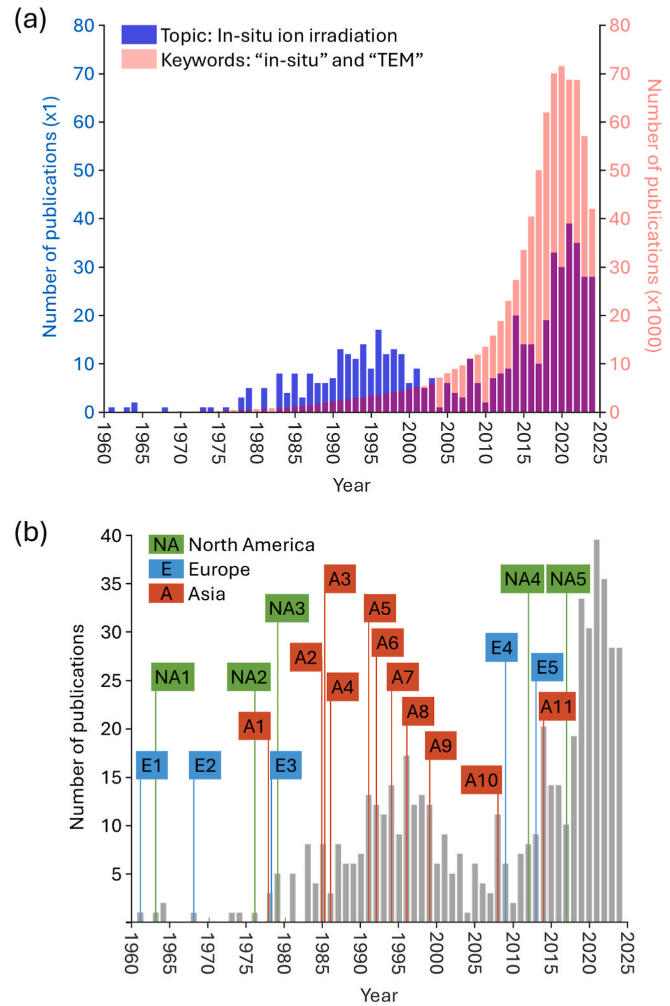
target environments. Even without the demands of scientific, technological and industrial applications, the study of the behavior and modification of materials under particle irradiation is an important subject of fundamental research. This is essential to understand why materials respond the way they do in such environments, to predict how technologies already in-service will perform in the future, to identify likely candidates for deployment in target applications and to engage in informed development of new material concepts.

It is the desire to explore materials on the atomic and nanoscale which leads us to reach for powerful characterization tools such as transmission electron microscopy (TEM) that allow us to probe and characterize the nano- and microstructure of materials both before and after exposure to irradiation. Conventionally, this can be performed by transferring the TEM sample to/from the irradiation facility (possibly repeatedly) or on TEM specimens extracted from bulk irradiated material. The former allows the same area(s) to be followed but may be limited by damage/contamination to the vulnerable TEM sample and the latter offers more versatility in terms of irradiation conditions whilst also reducing surface effects (should these be undesirable). Both approaches provide snapshots of the microstructure as it develops albeit with significant work required to capture each "Kodak moment" and important experimental parameters, most notably temperature, can be difficult to maintain throughout the process.

The evolution of a specimen due to radiation damage is a complex dynamic process in which multiple mechanisms compete with and/or complement each to determine the end state whilst also varying as functions of parameters such as bombarding particle mass/energy, dose, dose rate and temperature. Therefore, it is highly advantageous to be able to make observations and measurements to capture these phenomena in real time as they unfold. It was for these reasons that the technique of TEM with in-situ ion irradiation emerged in the 1960s [1] and remains an invaluable tool. With its ability to continuously observe the same area of the sample under controlled experimental conditions from the unirradiated to the end state, it can produce consistent datasets which track the entire story from beginning to conclusion.

The current review aims to provide not only an updated look at the construction and capabilities of TEM with in-situ ion irradiation facilities around the world but also the broader landscape in which they reside. This will be explored in terms of the collaborative links which connect them, the trends in the publications produced, the access routes to the instruments themselves and, importantly, the support of the underpinning highly-specialized user community. It is hoped that this will serve as a valuable resource for both those already familiar with the technique as well as those who may be newcomers seeking to engage for the first time. The interested reader may also wish to look back into the literature at previous publications which have incorporated reviews and histories of the development of this field [2–12].

The impact of these facilities to our fundamental understanding of radiation damage at the nanometer scale is immense. One metric to track this is the quantity of publications per year, as explored in Fig. 1. The blue/purple data in Fig. 1(a) plots the rapid rise of in-situ ion irradiation publications. While this is an impressive growth rate, it is still less than the expansion of the in-situ TEM field as a whole. This difference in growth rates has resulted in over three orders of magnitude more in-situ TEM general publications compared to those featuring in-situ ion irradiation specifically. The rapid recent expansion of in-situ TEM related publications more broadly can likely be attributed to the emergence of new fields including, but not limited to, in-situ electrical biasing, fluid flow and quantitative mechanical testing. A more nuanced examination of the in-situ ion irradiation publications list per year, see Fig. 1(b), indicates several interesting trends. First, there is a deviation not only from exponential growth, but a statistically relevant drop in publications in the 2000s. This trend can probably be attributed to the decreased funding in nuclear materials research in Western societies in the preceding years. The large upfront cost and time required for purchasing a TEM, an ion accelerator and the beamline to couple them



North America	Europe	Asia	
1) AECL	1) TIRL	1) Uni. of Tokyo	7) NIMS
2) Uni. of Virginia	2) AERE	2) CREIPI	8) TIARA
3) ANL	3) MIAMI	3) Hokkaido Uni.	9) Shimane Uni.
4) I³TEM	4) Gabriel Lippman	4) Hitachi	10) Wuhan Uni.
5) MIBL	5) JANNuS	5) Kyushu Uni.	11) Xiamen Uni.
		6) JAERI	

Fig. 1. Generated using publications pertaining to experimental results and developments of TEMs with in-situ ion irradiation capabilities identified using Google Scholar: (a) plot of the number of publications per year showing the growth of the discipline over time (left axis) and the growth of the overall in-situ TEM field using the keywords "in-situ" and "TEM" (right axis); and (b) the first publication in the dataset corresponding to each facility is indicated and these are grouped further by continent using color coding. See Table 1 for explanations of facility abbreviations. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

together demands that these new facilities be well supported. In addition, the operation of existing facilities requires extensive time, money and effort to maintain the relevance and the cutting-edge nature of world class infrastructure. Given the funding such research can attract, the periods of expansion can be correlated to the growth of the associated nuclear materials field in the relevant geopolitical regions. This is also highlighted by looking at the first recorded publication for each facility as indicated in Fig. 1(b). As a side note of caution and although every effort was made to avoid such pitfalls, it should be mentioned that, as an inevitable consequence of the natural evolution of scientific terminology, the primarily keyword-based analysis presented here can potentially overlook important early papers such as the first publication from the facility Orsay, France in 1983 [13]. Finally, this dataset also indicated that the facilities with the greatest impact on publications per year are the large facilities open to external users. Delving deeper into this, Section 4 of this review will highlight the collaborative user and operator community which underpins the outputs of TEM with in-situ ion irradiation facilities. With all that said, although the quantity of publications in the field is an easy-to-measure metric, it can be only a starting point to assess the impact and importance of this community.

2. Facility design

2.1. Coupling of TEMs with ion beam systems

Commercial TEMs in their standard off-the-shelf configurations are not intentionally designed for the incorporation of an ion beam and certainly not optimized for the specific requirements of performing real-time in-situ ion irradiation experiments. However, they do typically offer a range of vacuum ports envisaged for other activities which can be adopted for this application or the column can be modified (either by the original manufacturer or an adventurous microscopist) to provide the necessary access. Fig. 2 demonstrates four possible routes of ingress – each with its relative benefits and drawbacks:

- **Side entry ports:** Usually at the same vertical height as the sample, most TEMs will feature these for the mounting of devices which require access to the polegap of the objective lens such as analytical detectors. These therefore allow an ion beam to enter the column perpendicularly (or close to 90°) to the optical axis of the microscope. The port must be suitably orientated in the horizontal plane so as provide line-of-sight to the sample position taking the geometry of the sample holder into account; this is illustrated in Fig. 2 where the ion beam is incident normal to the x-axis (α-tilt) of the sample holder thus facilitating irradiation geometries which minimize the risk of shadowing. Consideration must be given to the strong magnetic field of the objective lens as the ions will be traveling normal to this and can therefore experience significant Lorentz forces. It may be possible to deflect the beam prior to entering the field to counteract this effect or the lens can be turned off during irradiation although obviously at the expense of the magnification and resolution of any concurrent TEM observation.
- **X-ray detector ports:** If present, these offer access above the objective lens but typically not line-of-sight to the sample position. Therefore, it is often necessary to implement in-column electrostatic deflection to bend the ion beam from its axis of entry onto a trajectory incident on the target. Whilst this introduces engineering challenges, especially given the limited space available to implement such a solution, it does have the advantage of offering the possibility of smaller angles between the ion and electron beams without necessarily having to modify the TEM itself. The schematic in Fig. 2 shows a simplified illustration of how such a solution might be integrated inside of the microscope.
- **Custom modification – through the polepiece:** Another option is to bore through the upper objective polepiece to provide a direct path to the sample position. Practically, this will require a circular

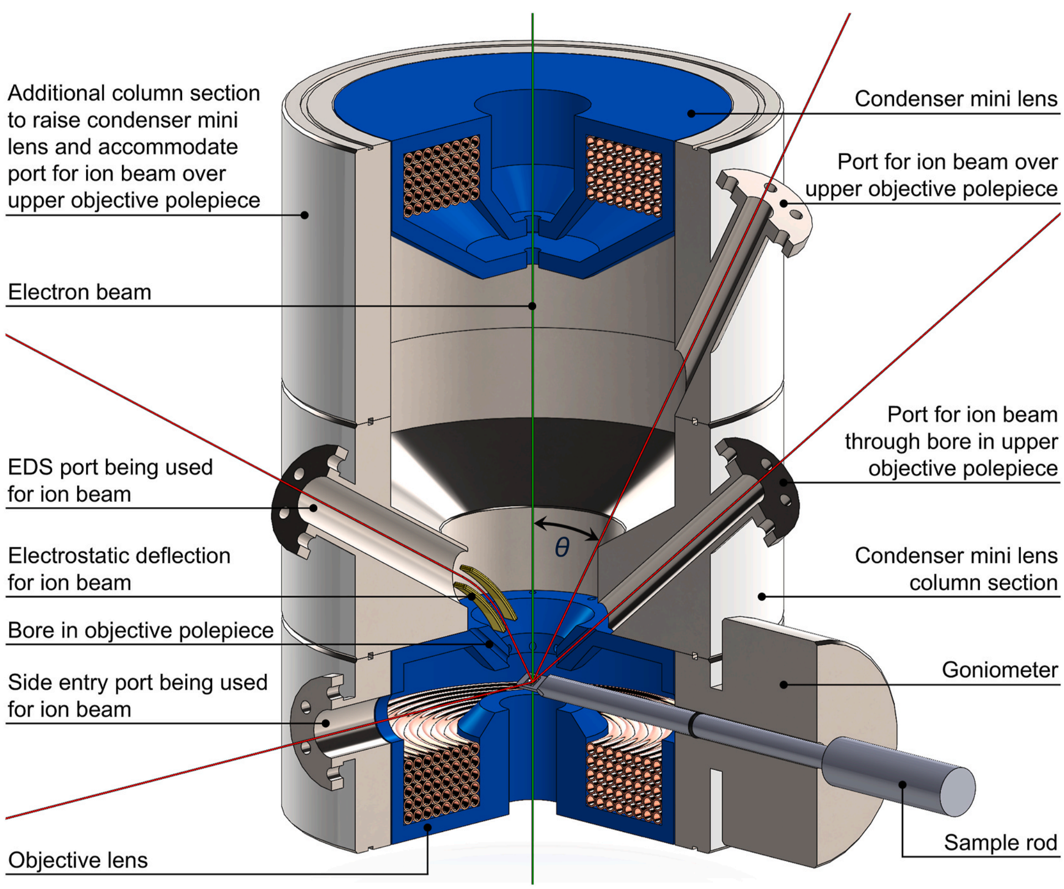


Fig. 2. Schematic showing four different approaches to introducing an ion beam into a TEM at various angles, θ, between the electron and ion beams: by repurposing the X-ray port combined with in-column electrostatic deflection ($\theta = 30^\circ$ as drawn); using a side entry port ($\theta = 90^\circ$ as drawn although smaller angles are possible by deviating from the axis of the port); via a custom port accommodated by an additional column section (thus raising the portion of the column above the objective lens and sample position starting from the condenser mini lens upwards) providing access over the top of the upper objective polepiece ($\theta = 30^\circ$ as drawn); and through a bore in the upper objective polepiece ($\theta = 50^\circ$ as drawn – note the circular pattern of these holes as discussed in the main text). The angle, θ, between the electron and ion beams is indicated for the example of a custom port bringing the ion beam in over the top of the upper objective polepiece.

pattern of such holes (as shown in Fig. 2) to reduce the possibility of introducing uncorrectable astigmatism or otherwise degrading the optical performance of the lens. Along with the addition of a custom port in the condenser section of the column, such modifications will likely be undertaken in collaboration with the TEM manufacturer. Like the repurposing of an X-ray detector port, this approach can deliver small angles relative to the electron beam.

- **Custom modification – above the polepiece:** By inserting an additional column section between the condenser and objective lenses, it is possible to expand this volume and create a direct-line-of-light to the sample position as can be seen in Fig. 2. As with adaptations to the objective lens polepiece described above, this modification is also a challenge perhaps best tackled by the TEM manufacturer as it will have implications not only for the electron optics but also the structure of the upper-portions of the instrument including the vacuum system and gun lift mechanism. Again, this offers the possibility of closing the angle between the ion and electron beams considerably.

As mentioned above, one of the important considerations involved in selecting the method of integrating a TEM with ion beamline(s) is the angle, θ, between the ion beam the optical axis of the microscope at the point of incidence with the sample. It can be advantageous in many, but not all, experiments to be working with as small a value of θ as possible for the following reasons:

- **Flexibility of TEM orientation:** Greater freedom in the choice of how the microscope is rotated (in the horizontal plane) can be achieved when designing custom ports which typically afford smaller angles. Conversely, if entering the column normal to the optical axis of the TEM through standard ports, then the possibilities are restricted by the combination of the predetermined geometry of those ports and the sample holder – together, these can limit the range of possible ion trajectories that are free of shadowing effects. For practical experimental reasons (such as ease of orientating the sample relative to the ion beam) entering normal to the X-axis of the sample holder is often preferable. Finally, flexibility in the orientation of the TEM can become important when other factors, such as structural constraints of the building, come into play.
- **Navigating the objective lens:** The magnetic field, B, in which the sample sits can exceed 1 T. Hence a transiting ion (of mass, m, charge, q, and velocity component, $v_\perp$, perpendicular to B) attempting to reach its target can experience a considerable Lorentz force, $F = Bqv_\perp$. However, $v_\perp$ reduces as the trajectory of the ions approaches the optical axis of the TEM thus lessening the effect for smaller values of θ. As the radius of centripetal motion is given by $r = mv_\perp/Bq$, the lighter and slower the ions then the greater the deflection they suffer and below a critical value of $mv_\perp/q$ it will no longer be possible to counteract this effect within the alignment capabilities of the ion beamline. Therefore, by reducing θ it is possible to get lower ion energy-mass-charge combinations on target.
- **Ion beam shadowing:** Whilst it is often possible to minimize the chances of ion beam shadowing by the holder and/or the sample itself (for example, by its own support grid) by using suitable sample tilting, the necessity for this is reduced at smaller values of θ. This then leaves greater flexibility in terms of sample orientation to address the further considerations detailed below.
- **Electron beam shadowing:** If it is necessary to tilt the sample to higher angles to avoid ion beam shadowing effects then consequently the risk of blocking the electron beam increases. This can reduce the observable proportion of the sample and thus potentially leading to important regions of interest becoming obscured.
- **Ion range:** Where the demands of an experimental design push the limits of the ion energies available, being able to irradiate parallel to the direction within the sample along which greatest ion range is required helps to maximize the possibilities. As described above, if the need to tilt the sample to avoid shadowing effects is reduced then the angular freedom available for the purposes of ion range extension are increased.
- **Effective sample thickness:** If the beamline-TEM geometry requires tilting the sample away from being normal to the optical axis of the microscope, the effective sample thickness can increase and thus reduce the useable electron-transparent regions (although not in all cases, with nanospheres being a notable exception).
- **Imaging conditions:** Similarly, if sample tilting is required to address the issues caused by larger values of θ as described above, this leaves a smaller useable angular range within which to obtain, for example, desired diffraction conditions (i.e., crystallographic orientations).

Whilst the above makes a case for the minimization of the angle between the ion and electron beams, it is not necessarily true that there are no advantages to entering at a large angle or even normal to the optical axis of the TEM column. As well as this being a potentially simpler implementation in terms of the required modifications to the instrument, it can have advantages such as allowing observation of the effects of an ion traveling across a sample. For example, with a suitable sample geometry such as that of a nanowire, it can be possible to observe effects on both the sides of entry and of exit of a transiting ion.

2.2. Selection of TEM

Whilst provision for suitable coupling of the microscope to the ion beam system is essential as explored above, there are some other important considerations when selecting a TEM for an in-situ irradiation facility.

- **Electron source and energy:** Most systems in operation today are based around 300 kV TEMs with LaB_6 sources as these give a suitable range of energies to help tackle a range of both electron-beam sensitive materials and thicker samples with good intensity levels for general imaging and analytical work. The additional brightness and coherence of field emission guns (FEGs) have generally not been employed as the additional purchase and running costs are not justified by the demands of the application but assessment has begun to change. Recent exceptions to this being the PIES facility (which uses a FEG for high-resolution characterization with in-situ secondary ion mass spectrometry (SIMS)) as well as the latest facilities at MIBL (zirconiated tungsten (ZrOW) thermal field emitter (TFE)) and at Xiamen University (FEG source).
- **Scanning and aberration-corrected TEM (STEM and ACTEM):** Whilst conventional TEM is most commonly used for imaging during in-situ ion irradiation experiments to allow continuous real-time observation, STEM adds enhanced imaging and analytical capabilities that can be employed before and after ion irradiation steps (or even during if sample stability allows). It is for these reasons that several facilities have selected (S)TEM instruments in order to provide access to both modes. Similarly, ACTEM can open structural characterization possibilities on the atomic scale but performing such imaging concurrently with in-situ ion irradiation would be highly problematic due to mechanically and thermally induced movement of the sample. In contrast to (S)TEM, no in-situ irradiation facilities have yet to go down the route of basing a system around an ACTEM primarily due to the costs of the instrument and service contracts not being justified by the potential experimental gains. However, another tantalizing possibility offered by ACTEM is that, rather than using the aberration correction primarily to obtain the highest spatial resolution possible, it can compensate for increased aberrations induced when widening the polegap of the objective lens to provide a larger working area for additional in-situ capabilities as discussed below.

- **Polegap size and access:** The mechanical options for the introduction of the ion beam into the microscope (as described above in Section 2.1) are a key factor when selecting a TEM. Beyond the interfacing of the beamline, space and access requirements are important considerations for additional in-situ environmental and characterization capabilities, as well as devices for ion beam monitoring and dosimetry. Although many capabilities can be incorporated into the TEM holder itself, there will always be experimental conditions (especially for exotic combinations) that necessitate additional access and space to accommodate the required instrumentation. For example, this may take the form of ports for laser heating or a sufficient polegap to allow the introduction of a Faraday cup directly before (or at) the sample position. A larger polegap is also generally an inherent property of high voltage electron microscopes (HVEMs) permitting greater flexibility for applications as currently being developed at the MQB-HVEM (see Fig. 4 and Table 1 for details of this facility).
- **Availability of sample holders:** The number of suppliers and the range of in-situ capabilities they offer continue to grow year-on-year. Despite this, not every holder is available for every model of TEM especially for some of the more niche applications from small manufacturers or new entrants to the market. Therefore, it is important to ensure that critical holders are available for any given microscope under consideration. Beyond the essential requirements at the outset of designing a facility, there is also the possibility to engage and collaborate with manufacturers to develop new capabilities or adapt existing technologies to new instruments so the absence of provision today should not be seen as an insurmountable barrier to what can be achieved tomorrow. As an example, a special low-profile holder was developed in collaboration with Gatan that has become a mainstay at the JANNuS-Orsay facility for in-situ ion irradiation experiments and more recently has also been deployed at MIBL (see Fig. 4 and Table 1 for details of these facilities).
- **Manufacturers:** Beyond the TEM models and variants/options offered alongside the accessories available from third-party suppliers, there are two further important factors when selecting a manufacturer. Firstly, are the possibilities for any specialist upgrades to the standard design that might be required; not every adaptation will be practical (or even within the bounds of current technology and, ultimately, physics) so it is essential to work with vendors to explore the possibilities. Secondly, service contracts should be considered not just from the point of view of the ongoing costs but also in terms of the provisions made to cover the modifications inherent to the instrument being incorporated into an in-situ irradiation facility.

2.3. *Ion beam systems*

Whether a dedicated ion beam system is being designed specifically for coupling to a TEM or a beamline is being chosen at an existing ion beam laboratory, the primary considerations are ensuring that the required range of ion species, energies and fluxes will be available at the sample position. As summarized in Table 1, nearly all facilities offer a combination of self and/or inert ions as well as light gas (H and He) ions primarily for nuclear material applications. This allows experiments to be performed with combinations of atomic displacements and implantation to explore neutron damage as well as the effects of nuclear transmutation reactions (fission, fusion and decay). Through the use of sequential and concurrent irradiations (as required and available), the consequences for microstructural evolution can be studied in combination, in isolation or in series. For example, the combinatory effects of the concurrent introduction of energetic primary knock-on atoms (PKAs) with light gas implantation can be compared to the development of pre-existing bubbles under heavy-ion irradiation or the formation of bubbles in pre-damaged material.

Ion energy ranges are generally selected to achieve desired depths and shapes of damage and/or implantation profiles. This may be to minimize/maximize ion retention or to achieve a desired ratio of damage-to-stopping as demonstrated in Fig. 3 for a 100 nm-thick TEM sample of silicon.[1] Due to the thin geometry of a TEM sample, it is typically possible to locate the damage (Bragg) peak beyond the sample thickness or similarly to select any desired portion of the implantation profile; the general rule being that higher energies will result in lower implantation and more-homogeneous distributions. Although beyond the scope of this review, it should be noted that additional considerations may come into play when varying ion energy, such as the rate of damage or implantation accumulation, the sputtering rate and the ratio of electronic to nuclear stopping.

3. Overview of facilities currently in operation

Each facility around the world has taken its own approach to the considerations outlined above in selecting the TEM, designing the ion beam system(s) and the integration of the two. These are illustrated in Fig. 4 and detailed in Table 1. Several trends have emerged over the last few decades with a greater adoption of dual ion-beamlines and a shift towards (S)TEM with analytical capabilities. Similarly, the increased demand for experiments that exceed temperatures of ~500 °C, coupled with the greater availability of sample holders offering such capabilities, has led to wider availability of such provisions.

4. Cross-facility collaboration and community

As the theory of electron and ion optics are well known, in principle an instrument such as those listed in Table 1 could of course be developed in isolation. However, in practice, the complexity of the integration is such that it can be highly advantageous to learn and benefit from the experiences (and mistakes!) of previous facility builds to potentially save time, money and sanity. Luckily, this is possible due to the open and integrated culture of the TEM with in-situ ion irradiation community including the regular conferences and workshops attended by both facility operators and users. The most focused and specialized of which is arguably the Workshop On Tem With In-Situ Irradiation (WOTWISI) series. The first WOTWISI was hosted at the University of Salford in 2008. Since then, it has been held in: USA (Sandia National Laboratories, 2011); Japan (Hokkaido University, 2013); France (Université Paris-Saclay, 2016); United Kingdom (University of Huddersfield, 2018); USA (University of Michigan, 2022); and China (Xiamen University, 2024).

Although WOTWISI is the only conference dedicated purely to the combination of electron microscopy with ion irradiation and its applications, specialist sessions can often be found at both electron microscopy events (for example, Microscopy & Microanalysis (M&M) and the European Microscopy Congress (EMC)) as well as accelerator conferences (such as the Conference for Accelerator Applications in Research and Industry (CAARI), the American Nuclear Society's Accelerator Application (AccApp), Ion Beam Modification of Materials (IBMM) and Radiation Effects in Insulators (REI)).

There are also many instances of joint research between facilities that couple TEMs and ion accelerators. One early example of such a collaborative experimental effort was by S.E. Donnelly (who went on to co-found the MIAMI facilities) et al. over multiple visits to the IVEM facility at Argonne National Laboratory (ANL) as highlighted in various works published in the 1990's [35–37]. That research studied the evolution of helium bubbles in thin gold and aluminum TEM samples under heavy-ion irradiation that resulted in understanding of the behavior of helium bubbles including their Brownian motion and the effects of direct displacement of the gas atoms. These insights would not have been possible without the combination of expertise in bubble evolution with the capabilities at the IVEM at that time. This cross-pond tradition continued in both directions, with J.A. Hinks visiting the I^{3}TEM facility at Sandia National Laboratories (SNL) to perform experiments studying

Table 1
Summary of technical specifications of the ten operational facilities around the world (high-angle annular dark-field (HAADF); focused ion beam secondary-ion mass spectrometry (FIB-SIMS); electron/ion beam induced current (EBIC/IBIC); photo/cathodoluminescence (PL/CL); quadrupole mass spectrometer (QMS); other abbreviations defined in the main text.

	Intermediate Voltage Electron Microscope (IVEM)	Multi-Quantum-Beam High-Voltage Electron Microscope (MQB-HVEM)	Ion Gun Direct-Coupled TEM Equipped with High-Resolution QMS	Joint Accelerators for Nanoscience and Nuclear Simulation (JANNuS-Orsay)	Parallel Ion Electron Spectrometry (PIES)	In-situ Ion Irradiation Transmission Electron Microscope (I^3TEM)	Microscope and Ion Accelerators for Materials Investigations (MIAMI-2)	High Fluence Irradiation Facility (HIT)	Michigan Ion Beam Laboratory (MIBL)	Xiamen Multiple Ion Beam In-situ TEM Analysis (XIAMEN)
Institution	Argonne National Laboratory	Hokkaido University	Shimane University	CNRS/IN2P3 and Université Paris-Saclay	Luxembourg Institute of Science and Technology	Sandia National Laboratories	University of Huddersfield	University of Tokyo	University of Michigan	Xiamen University
Country	USA	Japan	Japan	France	Luxembourg	USA	UK	Japan	USA	China
Established	1995	1998	1999	2007	2012	2014	2016	2016	2019	2019
TEM	Hitachi H-9000 NAR	JEOL ARM-1300	JEOL JEM-2010	FEI Tecnai G2 TWIN	FEI Tecnai F20	JEOL JEM-2100	Hitachi H-9500	JEOL JEM-2000FX	FEI Tecnai G2 F30 TWIN	FEI Tecnai G2 F30 TWIN
Electron source	LaB_6	LaB_6	LaB_6	LaB_6	FEG	LaB_6	LaB_6	LaB_6	ZrOW TFE	FEG
Electron energy	300 keV	800, 1000 and 1250 keV	80–200 keV	80–200 keV	80–200 keV	80–200 keV	60–300 keV	80–200 keV	80–300 keV	50–300 keV
Sample temp	−253 °C–1100 °C	Cryo to 500 °C	−253 °C–1300 °C	−173 °C–1300 °C	RT	−173 °C–1200 °C	−173 °C–1300 °C	RT to 900 °C	RT to 1300 °C	RT to 1200 °C
Beam angle (*θ*)	30°	46°	17°	68°	68°	90°	18.7°	30°	68°	68°
Analysis	EDS	Not applicable	Mass spectrometer for sample gas release	EDS, EELS, EFTEM and HAADF	EDS, EELS, EFTEM and FIB-SIMS	EBIC, IBIC, PL, CL and ACOM	EDS, EELS and EFTEM	Not applicable	EDS, EELS and EFTEM	EDS
Primary ion beam	H, inert gases and many elements from Al to Au	H_2, D_2, He, N_2, O_2, CO_2, Ar, Kr and Xe	Gases from H to Ar	65 elements	Ga	Most ions including H, He, Cu, Ni and Au	Most ions including H, He, Ne, Ar, Fe, Kr, Xe and W	Fe, Ni, Cr, Zr and W	Most ions including H, He, Ar, Fe, Ni, Kr and Ne	Most ions including H, He, Ar, Fe, Ni, Si, Kr and Ag
	50 keV to 1 MeV	25–300 keV depending on species	1–20 keV	10–470 keV	5–30 keV	46 keV to 48 MeV depending on species	5–350 keV (singly charged)	0.5–3 MeV	30–400 keV	30–400 keV
Secondary ion beam	He	N_2, O_2, CO_2, Al, Si, Ar, Ti, Fe, Ni, Cu, Kr, Xe, W and Au	Not applicable	35 elements	Not applicable	Gases including H, D and He	H and He	Not applicable	H, He, NH and O	H_2 and He individually or simultaneously
	10 keV–20 keV	25–400 keV depending on species	Not applicable	Few hundred keV to 6 MeV	Not applicable	100 eV to 20 keV	1–20 keV	Not applicable	20 eV to 50 keV	10–50 keV
Ion-TEM interface	Above upper objective polepiece via custom port	Through upper objective polepiece via custom port	Above upper objective polepiece via EDS port	Between objective polepieces via side port	Between objective polepieces via side port	Between objective polepieces via side port	Above objective polepiece via custom port	Above objective polepiece via custom port	Between objective polepieces via side port	Between objective polepieces via side port
Reference (s)	[3,14,15]	[16]	[17,18]	[2,19–23]	[24,25]	[26–28]	[29]	[30]	[31–33]	[34]

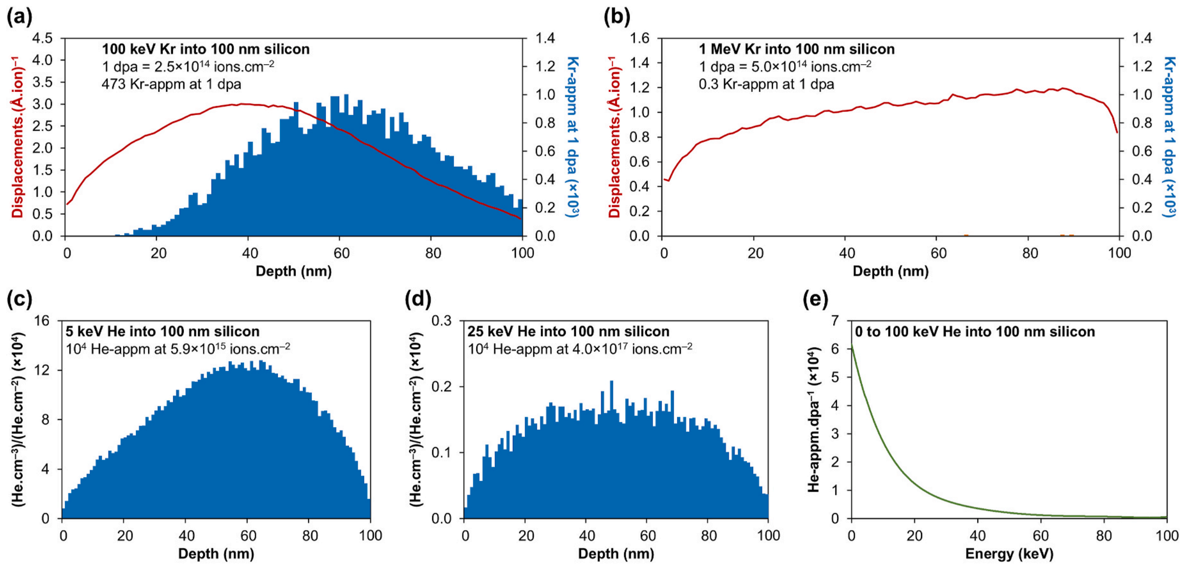


Fig. 3. Illustrative examples[11] of how ion energy can be tuned to achieved desired effects in a 100 nm-thick TEM sample of silicon: (a) combined damage and implantation for 100 keV Kr ions; (b) damage with minimal implantation for 1 MeV Kr ions; (c) maximizing implantation with 5 keV He ions; (d) smoothing of an implantation profile at the expense of implantation rate with 25 keV ions; and (e) tailoring of implantation-to-damage ratio for He ions (He-appm.dpa^{-1}). As functions of depth into the target, the solid red lines represent the atomic displacements induced (per ion per unit length) and the blue bar graphs represent ion stopping (see axes for relevant units). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

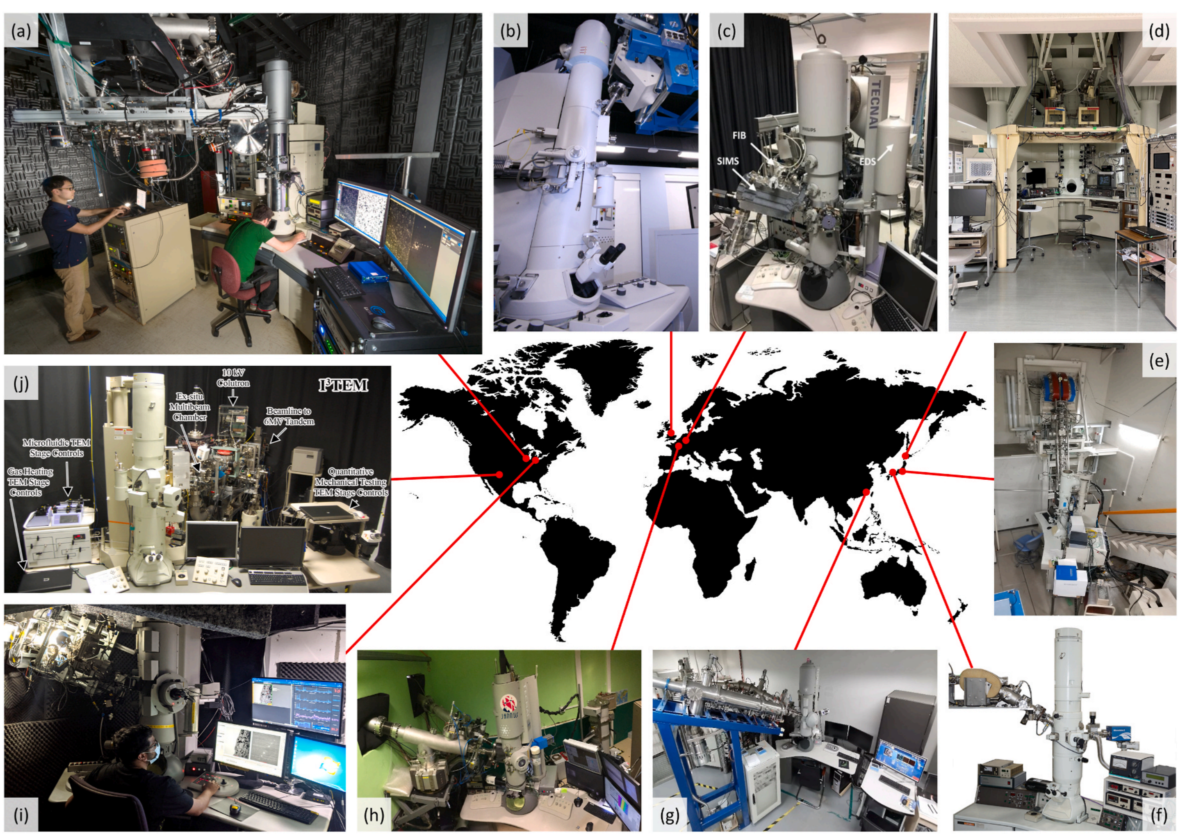


Fig. 4. TEM with in-situ ion irradiation facilities around the world: (a) IVEM at Argonne National Laboratory, USA; (b) MIAMI-2 at the University of Huddersfield, UK; (c) PIES at the Luxembourg Institute of Science and Technology, Luxembourg; (d) MQB-HVEM at Hokkaido University, Japan; (e) HIT at the University of Tokyo, Japan; (f) Shimane University facility, Japan; (g) XIAMEN at Xiamen University, China; (h) JANNuS-Orsay at the MOSAIC platform of IJCLab jointly run by CNRS/IN2P3 and Université Paris-Saclay, France; (i) MIBL at the University of Michigan, USA; and (j) I^{3}TEM at Sandia National Laboratories, USA. See Table 1 for further details.

the nanoscale impact of crystallographic channeling of ions in Au nanorods exposed to 1.7 MeV Au irradiation [38]. Observation of the dynamic evolution of the cratering and ejecta blanket enabled by in-situ TEM observation coupled with molecular dynamic (MD) simulations by K. Nordlund at the University of Helsinki provided a deeper understanding into the governing mechanisms associated with nanoscale sputtering in this system. Shortly after the commissioning of MIAMI-2, S. A. Briggs and K. Hattar from SNL traveled to the University of Huddersfield to study the impact of cryogenic temperature, hydrogen gas exposure and helium implantation on the evolution in Pd foils in an effort to better understand He bubble evolution in aged palladium tritide [39]. Sometimes, instruments from multiple institutes are utilized in concert as was the case in the study by F. Fortuna, M.-O. Ruault et al. utilizing both the JANNuS-Orsay and IVEM facilities to understand the precipitate nucleation mechanisms in silicon under various radiation conditions [40]. Other collaborations have focused directly on the development of TEM with in-situ ion irradiation infrastructure. One such recent example was between the MIBL, IVEM, I^3TEM and JANNuS-Orsay facilities to conduct commissioning experiments that integrate machine learning (ML) and automated feature detection technologies such as the Theiascope systems and ANL-developed ML to evaluate the feasibility of exploiting such technologies in this field. These examples highlight the possibilities for novel scientific discoveries in everything from nanoparticle stability to hydrogen isotope storage and transport – all enabled by the collaborative nature of the community.

Whilst the aforementioned examples of collaborations between facility teams seeking complementary capabilities have been highly fruitful, it is even more common for users to utilize multiple facilities to accumulate complementary in-situ datasets to better explore the underlying mechanisms at play under extreme conditions. As a user of multiple TEM in-situ ion irradiation facilities, O. El-Atwani has demonstrated across several publications how a deeper understanding of a material system can be achieved by merging capabilities in this way. An example of such results is work on ultrafine grained and nanocrystalline tungsten utilizing both the MIAMI and I^3TEM facilities; this combination permitted a wide range of He energies and tilt ranges that would not be possible utilizing just one of these and thus provided greater insights into the role of grain boundaries as helium sinks [41]. In a similar fashion, C. Sun accessed the different TEM imaging and diffraction techniques possible at the I^3TEM and IVEM facilities to gain a deeper understanding of L12-structured γ' Ni_3(Al,Ti) precipitates in nickel superalloys under heavy-ion irradiation [42]. That study found, under the conditions studied, that above 18 dpa the γ' precipitates and matrix intermix providing a pathway to local control of the crystallographic long-range ordering and hence properties of the material. These two examples of the joint research done across instruments provide a glimpse of the collaborative nature of the field and the complementary nature of the various facilities.

The most common interaction between the facilities listed in Table 1 with the broader nuclear materials and ion beam modification communities is through engagement facilitated by national or international user access programs. These are very much connected to the vision and priorities of the host country, funding agencies, and local institutions. As a result, the nature, duration and speed of access to a TEM with in-situ ion irradiation can vary. Taking the USA facilities as an example, over the last two decades there has been a transition in the support of these from the Department of Energy (DOE) Basic Energy Science (BES) to the DOE Office of Nuclear Energy (NE) through the Nuclear Science User Facilities (NSUF). The NSUF has been highly successful at providing free-at-the-point-of-use access to users and funding to support the three facilities (IVEM, MIBL, and I^3TEM) at various levels. As a result, NSUF has been associated with most of the publications from these USA facilities over the last decade. Similarly in France, the Etude des Matériaux sous IRradiation et Analysis (EMIR&A) network of research infrastructure [43] includes ion and electron accelerators (dedicated to irradiation and analysis of molecules and materials) providing user access to the benefits of in-situ ion irradiation research across Europe and beyond. Access to the JANNuS-Orsay facility is also available through European funding programs such as Horizon. Since all the facilities featured in Fig. 4 and listed in Table 1 require extensive expertise and support to be kept active, it is important that adequate and long-term financial resources be maintained. Where advantageous, the user-access program model can provide facility scientists the opportunity to focus on instrumentation, maintenance and development to enable the experiments for the userbase who in turn are well placed to contribute with their scientific questions, novel materials and data analysis techniques. This symbiotic relationship can greatly increase the productivity of TEM facilities with in-situ ion irradiation beyond that which could otherwise be achieved within a single research group.

5. Future directions

Growth within the TEM with in-situ ion irradiation community has been strong over the last decade with four new facilities (MIAMI-2, HIT, MIBL and XIAMEN) brought into full operation since 2016. Through the community and exchange of knowledge, the burden of designing, installing and commissioning such facilities has been reduced with lessons learned and shared with each build. Similarly, by communicating capabilities through channels such as WOTWISI and review papers [3,4,7–10,12], gaps and potential synergies can be filled and realized, respectively. Examples of such are the incorporation of an environmental TEM (ETEM) system into MIAMI-2 and the chemical mapping capabilities added to the MIBL facility. At the time of writing the current review, there are two further facilities under development at the University of Tennessee, Knoxville (UTK), USA and Queen's University, Canada.

The in-situ TEM irradiation facility at the University of Tennessee, Knoxville (UTK) is currently being added to the Tennessee Ion Beam Materials Laboratory (TIBML). The TIBML operated for over a decade as a world class Ion Beam Analysis (IBA) facility centered around a 3 MV NEC Tandem Pelletron accelerator under the directorship of W.J. Weber and management of M.L. Crespillo. Upon the retirement of W.J. Weber, K. Hattar has transitioned from his previous role developing the I^3TEM facility at SNL to be the new director of TIBML. As part of this handover, TIBML will be adding a 300 kV NEC implanter, two high-flux low-energy ion sources with a number of beamlines and various in-situ capabilities. This includes the addition in September 2023 of a JEOL JEM-2100Plus STEM with modifications for time resolved microscopy from IDES Inc. This 200 kV LaB_6 instrument will incorporate modifications for increased brightness, tilt range, laser irradiation along the electron path and an ion port normal to the electron beam. In addition, the TIBML STEM facility will incorporate precession electron diffraction (PED) complemented by NanoMEGAS ASTAR capabilities to provide automated crystallographic orientation mapping (ACOM) and other 4D STEM techniques. Furthermore, state-of-the-art ML and edge computing from Theia Scientific will provide increased robustness and speed of video analytics. The status of the development of this new facility as of May 2025 can be seen in Fig. 5(a). At the time of writing, the STEM is installed and operational awaiting the addition of a Gatan ClearView camera and the necessary IDES modifications to the column. Currently, three phases of coupling ion beams into the STEM are planned. The first will incorporate the addition of a differentially pumped 20 kV Non-sequitur Technologies Model 1402 Ion Gun directly to the STEM. The second phase planned for the facility will connect the tandem accelerator as illustrated in Fig. 5(b). Finally, the third phase of development will incorporate the 300 kV ion implanter. Once all three are

[1] Calculations were performed using the Stopping and Range of Ions in Matter (SRIM-2021) Monte Carlo computer code [64] running in "Monolayer Collision Steps" mode with the default values and settings of the software.

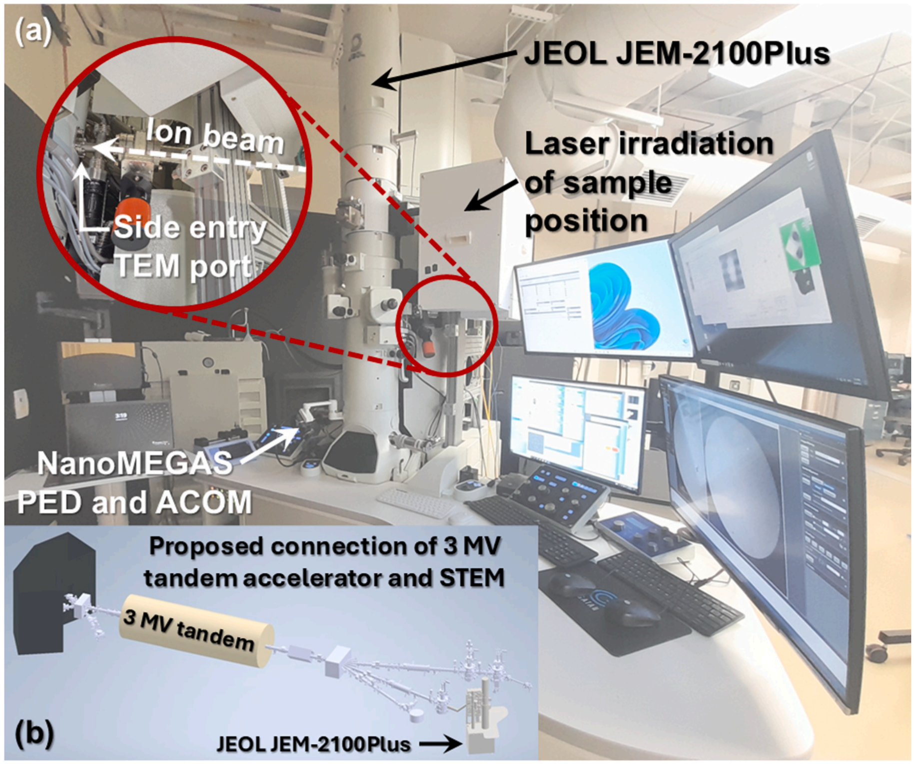


Fig. 5. The development and evolution of the TIBML which will couple a JEOL JEM-2100Plus STEM to multiple ion sources including a 3 MV tandem accelerator: (a) status of the facility as of May 2025; and (b) schematic of the potential ion beamline configuration being considered.

successfully implemented, it will be possible to select from a wide range of ion species and several orders of magnitude of ion energies and fluxes. The ambitious vision of the TIBML STEM facility, as with others previously established by the community, will take several years of development to realize.

A new multi-ion-beam TEM with in-situ irradiation facility is also being developed at Queen's University in Ontario, Canada with the design process and procurement of major components well underway at the time of writing. This instrument will be a FEG (S)TEM connected to both heavy-ion and light-ion beamlines. For these purposes, a new heavy-ion accelerator is being acquired to couple with the TEM; however, the facility will be co-located on the site of the existing 4 MV tandem ion source at the Queen's Reactor Materials Testing Laboratory [44].

It is expected that the current trend for combined environments will continue into the future driven by improvements in both the hardware and software. One of the areas of rapid growth in the electron microscopy field is in the development of TEM accessories that enable ever more precise control of the environments that samples are exposed to during in-situ observation. This includes moving beyond qualitative straining and sample-wide temperature control to applying quantitated loads and localized induced heating. In the later part of the 20th century, simultaneous ion irradiation, heating and straining became possible [45]; however, these studies were restricted in terms of temperature control due to the stage technology of the day and were limited to user-driven pulsed straining. This is no longer the case thanks to recent advancements in quantitative in-situ heating and straining. The development of in-situ TEM microelectromechanical systems (MEMS including microfluidics) and laser-based [28] heating permits much greater control both spatially and in terms of heating/cooling rates. As with any technique, both laser-based heating and MEMS introduce new challenges such as accurately determining local temperatures [46] and the potential to destroy samples with greater speed and efficiency than ever before possible [47]. New holder designs must also be vetted for deployment in irradiation environments; for example, MEMS devices can experience hazards under exposure to ion beams such as radiation damage altering the resistivity (and thus calibration) of embedded resistive heating elements or MEMS chips themselves can undergoing phase transformations leading to catastrophic failure modes. Similarly, recent developments in quantitative mechanical loading and incorporation of such into in-situ ion irradiation experiments permit testing of high-cycle fatigue [48], high temperature creep [49,50] and even irradiation-induced creep [51] in these environments. Of course, all of these advancements bring with them new challenges in addition to the baseline perils of working with thin films, vacuum and electron/ion beam exposure.

Advancements have not just been limited to modifications to peripherals such as sample holders, but are now impacting well inside the heart of the TEM. An example of this is the creation of a user-adjustable polegap (UAP) in work led by L. Jones at Trinity College Dublin which has been installed on the I^{3}TEM as illustrated in Fig. 6. In principle, this permits the sample to access extreme angles of tilt with a polegap of up to 6.5 mm; advantageous for any facility but particularly so for those utilizing a side entry port to introduce an ion into the TEM. The same polepiece can also shrink its polegap to 1.5 mm to allow high resolution imaging on the same sample all whilst maintaining the vacuum [52]. For example, a Gatan double-tilt heating holder Model 652 can be α-tilted to ~51.6° within a standard JEOL high-contrast pole piece (4.5 mm polegap) which is theoretically increased to ~74.5° at the widest polegap achievable using the UAP in the I^{3}TEM. The polegap is adjustable by the user (without opening the microscope) by coupling an electric motor to a gear system in the polepiece via a port in the objective lens column section – thus enabling microscope functionality to be changed within minutes.

As in-situ irradiation TEMs are coupled with increasing numbers of environments and analytical techniques, ever greater expertise is required of experimentalists in conjunction with further advancements in automation and integration of instruments. Given the wide range of

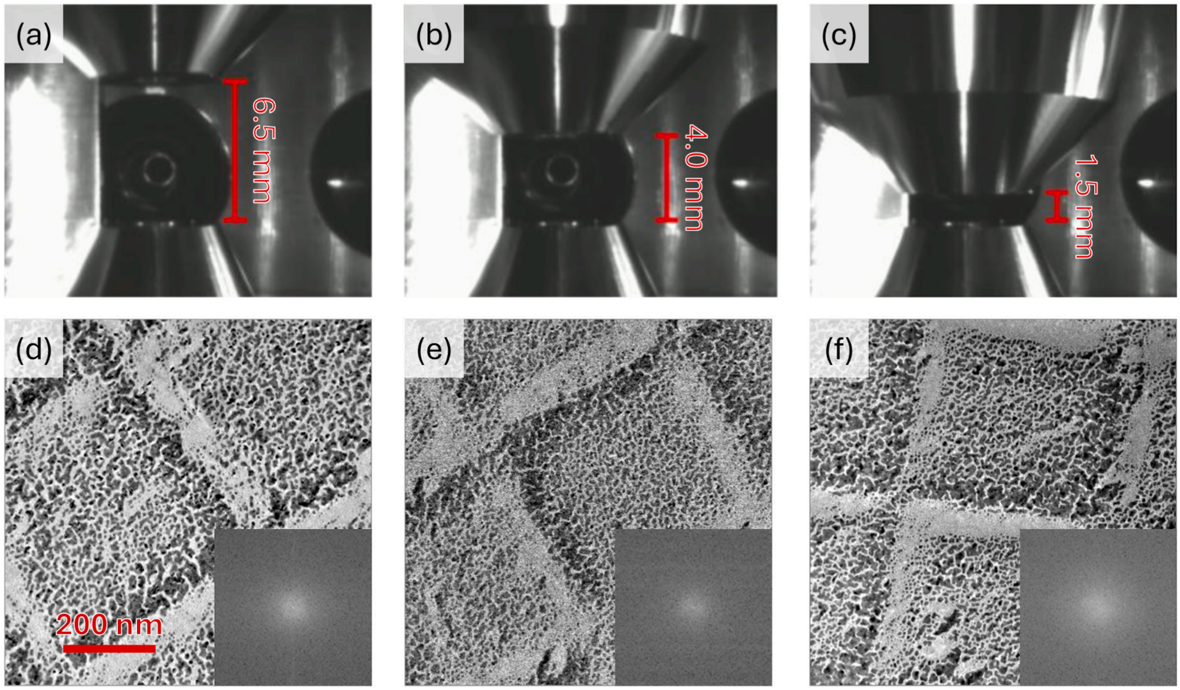


Fig. 6. The incorporation of an adjustable polepiece into the I^3TEM facility at SNL. The polegap can be readily adjusted from (a) 6.5 mm through (b) 4.0 mm to (c) 1.5 mm within minutes. The associated bright field images and fast Fourier transforms (FFTs) of a magnification-calibration grid for these polegaps can be seen in (d–f), respectively.

skills and knowledge already required to successfully execute complex experiments, such advancements in equipment are becoming ever more critical. Already there are both commercial and open-source options to assist in many basic of the operations of TEMs, stages and even accelerators; however, it can be expected that this trend will drive rapid developments powered by advancements in control systems and ML algorithms. For example, new high-speed controls for electron beams [53] can enable better distinguishment of the impacts of electrons from those of ion dose during in-situ irradiation experiments.

Another rapidly developing area of electron microscopy over the last several decades has been the advancement in electron sources, optics and detectors improving both the spatial and temporal resolution. The increases in spatial resolution provided by ACTEM has allowed polegap widening to accommodate a greater variety of in-situ devices and environments [54]. Similarly, new electron optics including dynamic TEM (DTEM) [55] and ultrafast TEM [56] have taken temporal resolution into the nanosecond and picosecond ranges for the capture of both destructive and reversible phenomena. These limits are now being pushed into the attosecond regime [57] and such advancements in spatial and temporal resolution continue to close the gap between MD simulations and experiments.

Increased resolution and faster image capture means larger and greater numbers of images to process. This exponential increase in dataset size, especially over the last two decades, means that data analytics has been under pressure to keep pace. An emerging approach is ML for applications such as the counting of radiation damage artefacts. The demand for such capabilities has given rise to new ventures such as Theia Scientific who have undertaken extensive testing in collaboration with the MIBL facility [58]. Such analysis can save days to months in the time required to go from data collection to publication of a successful in-situ experiment. In addition, the use of known and validated algorithms for data analysis (accompanied with appropriate reporting of the techniques used) will hopefully eliminate, or at least help to quantify, systematic errors which can arise from variability in the judgement of different individuals when assessing the same micrograph. This can lead to differences in the defect distributions identified between scientists from different countries, institutes and even within the same research group [59]. Maybe most importantly, it will help in real time to determine how an experiment is progressing, permitting adjustments to conditions to be made on-the-fly as might be required. Such analysis is illustrated schematically in Fig. 7(a) along an example of how microstructural phases can be quickly characterized using ML-based approaches in Fig. 7(b) [60]. These approaches combined with ACOM mapping of phases can allow rapid characterization of unexpected phases such as those that have been reported to form under ion irradiation of material systems including HCP Au and Ni [61,62]. Finally, the development of efficient and thorough characterization of spectra from energy dispersive X-ray spectroscopy (EDS), electron energy loss spectroscopy (EELS) and energy-filtered TEM (EFTEM) collected before, during or after in-situ ion irradiation is another tantalizing development. An example of this can be seen in the chlorine implantation EDS analysis shown in Fig. 7(c) [63]. Such approaches offer ways to track radiation-induced segregation via chemical mapping and/or local bond structure evolution. The ability to perform rapid characterization of defect distribution, diffraction patterns and spectra will greatly increase the speed and depth at which in-situ ion irradiation datasets can be processed.

With the introduction of seven new facilities in the last two decades, and two further systems under construction, combined with recent developments in electron and ion optics control, the expansion of in-situ environments now accessible and advancements in analysis techniques together provide a promising future for the TEM with in-situ ion irradiation community. As demonstrated in Fig. 1, the field is expanding rapidly thanks both to the growing demand for fundamental understanding of irradiation mechanisms as well as the increasing accessibility and capabilities of such facilities.

6. Summary

As this review has shown, the continuing demand from applications coupled with advancements in materials science is driving the need for investigation and assessment of samples under exposure to complex and

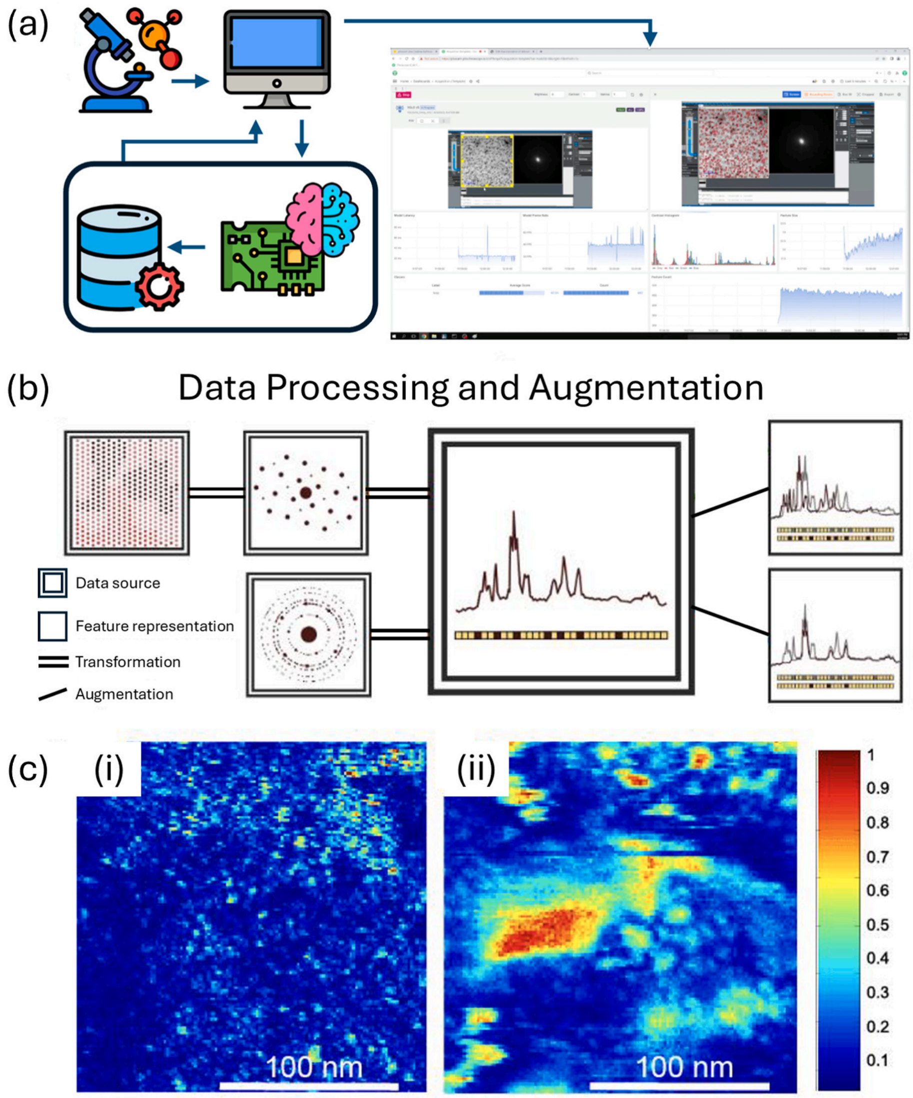


Fig. 7. The potential for real-time data processing illustrated for image, diffraction and spectral analysis: (a) ML-based analysis by Theia Scientific allowing real-time tracking of radiation induced defects such as dislocation loops or bubbles with an example shown for loop splitting [58]; (b) uniform feature vectors from diffraction data can be utilized in modular neural networks and combined with chemical information to improve the ability of a model to predict crystal structures [38]; and (c) Automated eXpert Spectral Image Analysis (AXSIA developed at SNL) ML-based analysis enables spatial identification of Cl component from plan-view Al thin film samples implanted with 35 keV Cl^{+} to (i) 5×10^{16} $Cl.cm^{-2}$ and (ii) 7×10^{16} $Cl.cm^{-2}$ [63].

extreme environments. This need is ideally met by TEMs with in-situ ion irradiation capabilities supported by a community of international researchers. Such instruments serve to provide fundamental understanding of the mechanisms at work in these coupled environments on the nano- and microscales; this is essential for the testing of existing materials, development of new solutions and the experimental-validation of modelling work.

Additionally, the prospects and opportunities for coupling these facilities with the latest scientific apparatus, instrumentation and techniques is opening new possibilities through the exploitation of technologies such as MEMS and microfluidic systems as well as advanced control systems and ML-based analysis tools. Maximal benefit from the implementation of such solutions can be achieved only if supported by an international community of collaborative researchers and technologists; fortunately, this is a resource in which the field of TEM with in-situ ion irradiation finds itself well endowed.

CRediT authorship contribution statement

Khalid Hattar: Conceptualization, Writing – original draft, Writing – review & editing. **Rishabh Kothari:** Data curation, Formal analysis, Writing – review & editing. **Wei-Ying Chen:** Writing – review & editing. **Mark R. Daymond:** Writing – review & editing. **Santhana Eswara:** Writing – review & editing. **Kevin G. Field:** Writing – review & editing. **Aurélie Gentils:** Writing – review & editing. **Abe Hiroaki:** Writing – review & editing. **Lewys Jones:** Writing – review & editing. **Arakawa Kazuto:** Writing – review & editing. **Mitsutaka Miyamoto:** Writing – review & editing. **Guang Ran:** Writing – review & editing. **Tamaki Shibayama:** Writing – review & editing. **Christopher M. Smyth:** Writing – review & editing. **Gary S. Was:** Writing – review & editing. **Jonathan A. Hinks:** Conceptualization, Writing – original draft, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal

relationships which may be considered as potential competing interests: Kevin Field reports a relationship with Theia Scientific, LLC that includes: equity or stocks. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

No new data were used for the research described in this review article.

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