ANL/MSD/CP--86961
CONF-951155--65

# NEW INSTRUMENTATION IN ARGONNE'S HVEM-TANDEM FACILITY: EXPANDED CAPABILITY FOR IN SITU ION BEAM STUDIES*

Charles W. Allen, Loren L. Funk and Edward A. Ryan
Materials Science Division
Argonne National Laboratory
Argonne, IL 60439

RECEIVED
FEB 08 1996
OSTI

November 1995

The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. W-31-109-ENG-38. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes.

**DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Submitted to the Materials Research Society (MRS) 1995 Fall Meeting, November 27–December 1, 1995, Boston, MA.

*Work supported by the U. S. Department of Energy, BES-Materials Sciences, under Contract W-31-109-Eng-38.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

MASTER

# NEW INSTRUMENTATION IN ARGONNE'S HVEM-TANDEM FACILITY: EXPANDED CAPABILITY FOR IN SITU ION BEAM STUDIES*

CHARLES W. ALLEN, LOREN L. FUNK and EDWARD A. RYAN
Materials Science Division, Argonne National Laboratory, Argonne, IL 60439

## ABSTRACT

During 1995, a state-of-the-art intermediate voltage electron microscope (IVEM) has been installed in the HVEM-Tandem Facility with in situ ion irradiation capabilities similar to those of the HVEM. A 300 kV Hitachi H-9000NAR has been interfaced to the two ion accelerators of the Facility, with a spatial resolution for imaging which is nearly an order of magnitude better than that for the 1.2 MV HVEM which dates from the early 1970s. The HVEM remains heavily utilized for electron- and ion irradiation-related materials studies, nevertheless, especially those for which less demanding microscopy is adequate. The capabilities and limitations of this IVEM and HVEM are compared. Both the HVEM and IVEM are part of the DOE funded User Facility and therefore are available to the scientific community for materials studies, free of charge for non-proprietary research.

## INTRODUCTION

Since 1961 when Pashley and Presland [1] reported observations of unintentional in situ ion damage in Au in an 80 kV TEM, involving negative ions from the microscope's electron source, the area of in situ TEM studies of ion irradiation effects has become well established for both fundamental and applied irradiation effects research, especially since the late 1970's. Today there are more than a dozen facilities at which one or two ion accelerators or ion sources are interfaced to 100–1250 kV TEMs, most of which are in Japan. Recently Ishino [2] has presented a brief historical overview of this type of instrumentation as it has evolved over the past nearly quarter century.

The instrumentation architecture of such a facility is straight forward in concept and involves only relatively minor modification of the TEM in the vicinity of the objective lens by the TEM manufacturer to accomodate the introduction of the ion beam. JEOL, Hitachi and Philips all have some experience in this regard. The original concept sketch of the HVEM-Tandem Facility is shown in Fig. 1 which depicts the tandem accelerator and the ion beam interface to the HVEM with its double-tanked Haefely electron accelerator system. In the modification of the TEM for such a facility, the major design decision concerns the angle between the electron and ion beams, which in the various installations has ranged from 30 to almost 90 degrees. For simultaneous plan view irradiation and TEM observation, the practical limit, however, is about 50 degrees with the specimen tilted toward the ion beam half of that limit or more during irradiation to avoid shadowing by the specimen holder. The second major concern which must be dealt with in the design phase is the minimization of mechanical vibration transmission from the ion beamline to the TEM if good spatial resolution for imaging is to be achieved. The third important factor is the manner in which the ion dosimetry is conducted. In the majority of installations, this is accomplished by replacing the specimen holder with a holder in which the specimen position is occupied by a miniature Faraday cup. This precludes the possibility of

---

* Work supported by the U. S. Department of Energy, BES-Materials Sciences, under Contract W-31-109-Eng-38.

au

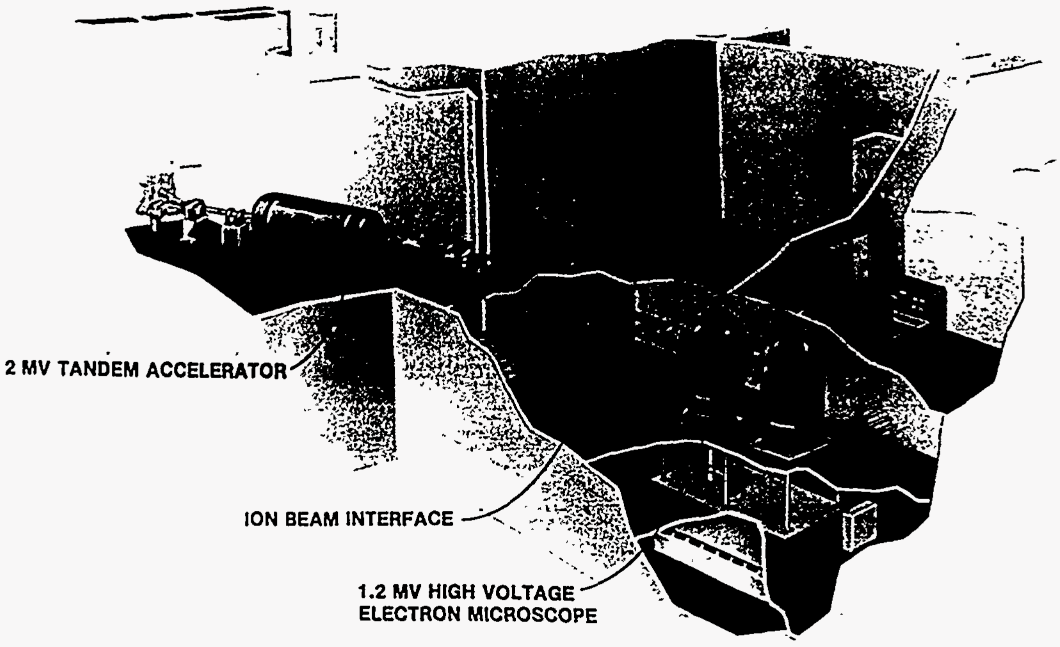

Fig. 1. Original concept sketch of the HVEM-Tandem Facility (1979).

checking the dosimetry during an experiment without withdrawing the specimen, which, especially for cryogenic experiments, may be out of the question, and at best, very inconvenient. An alternative solution is a moveable Faraday cup permanently installed inside the microscope column, which can intercept the ion beam path just prior to the TEM objective lens [3]. Each of the world's facilities in operation in 1993 has been briefly described and referenced [4]. The techniques of in situ TEM studies in ion beam research have proven to be efficient and cost effective and even essential to many cryogenic studies in order to avoid the mitigating effects of warming before TEM observation. The purpose of this paper is to summarize a number of salient technical aspects of the newly configured HVEM-Tandem Facility for the benefit of the community of users and potential users .

## INSTRUMENTATION OF THE HVEM-TANDEM FACILITY

The HVEM-Tandem Facility now consists of two transmission electron microscopes, (1) a modified AEI HVEM (EM 1200), installed in 1979, with accelerating voltages ranging continuously from 0.1-1.2 MV, and (2) an Hitachi H-9000NAR intermediate voltage electron microscope (IVEM), with accelerating voltages from 100–300 kV, installed in 1995. Both microscopes are interfaced to a 2 MV tandem ion accelerator and a 0.65 MV ion implanter. The tandem is somewhat unique in that it incorporates a positive ion source at its center, allowing positive ions injected at this point to be accelerated through the full potential drop of the machine, resulting in relatively large beam currents of species which are difficult to produce as negative ions (noble gases, for example). This combination of instrumentation, ion accelerators interfaced to an HVEM, offers unique capability for a wide range of in situ experiments involving electron irradiation, ion irradiation or ion implantation with simultaneous electron microscopy and diffraction. By the addition of the IVEM in 1995, with its vastly improved spatial resolution for imaging, larger useable magnification range and brighter electron source, this experimental capability is significantly expanded. Selected specifications for the IVEM are shown in Table 1.

Table 1. Selected Hitachi H-9000NAR (ANL) Specifications/Operating Conditions

| | |
|---|---|
| Accelerating Voltages | 100–300 kV |
| Specimen Chamber Vacuum | $<4\times10^{-7}$ Torr |
| Spherical Aberration Coefficient ($C_s$) | 2.8 mm (HVEM: 13.2 mm) |
| Objective Lens Pole Piece Gap | 11 mm (HVEM: 18 mm) |
| Goniometer Tilts | ±45° (Eucentric X-Axis); ±30° (Y-Axis) |
| Minimum Spot Size at 300 kV | ~25 nm (HVEM: 0.5 μm) |
| Magnification Ranges | |
| Low Mag | 200–500 X |
| Selected Area Mag | 4–300 kX |
| Zoom | 1–1,000 kX |
| Image Resolution | |
| Guaranteed Point-to-Point Lattice at 300 kV; Ion Beamline Disconnected | 0.25 nm |
| Guaranteed Line at 300 kV; Ion Beamline Disconnected | 0.14 nm |
| (Guaranteed resolution also achieved with ion beamline connected) | |

| | |
|---|---|
| Recording Capabilities | |
| Film (8 cm x 10 cm) | SO-163 (38 sheets) |
| Video Capabilities: | |
| Gatan Model 622 Image Intensified Camera on Column Axis | |
| Avio Image S-II Real Time Processor (Also serves HVEM) | |
| VHS Video Tape Format (Also serves HVEM) | |

Specimen Holders

- Ambient Single Tilt Holder (Hitachi)
- Ambient Double Tilt Holder (Hitachi)
- Heating Double Tilt Holder: 300–1300 K (Gatan)
- Double Tilt Helium Holder: 15–300 K Continuous (Spring 1996; Oxford)
- Faraday Cup Holder (ANL)

| | |
|---|---|
| Ion Beam | |
| Angle Between Incident Electron and Ion Beams | 30° |
| Ion Beam Diameter at Specimen Position | ~1.5 mm |
| Ion Irradiation Conditions (Limited by HVEM Wall Bending Magnet) | |
| 650 kV Accelerator—All ions to 450 kV | |
| Tandem—$\mathrm{He}^+$ to 2 MV; $\mathrm{Ne}^+$ to 1.8 MV; $\mathrm{Ar}^+$ to 0.9 MV; $\mathrm{Kr}^+$ to 0.45 MV | |
| Ion Beam Dosimetry | |
| Skim Cup/Faraday Cup System in IVEM Column; Faraday Cup Holder | |

In Table 1 several specifications for the HVEM have been included for comparison. The spherical aberration coefficient for the objective lense of the HVEM is 13.2 mm, nearly five times larger than that of the IVEM. This rather large value results from the fact that the gap of the objective lens pole piece of the HVEM is 18 mm (IVEM: 11 mm). The resolution of the HVEM is further compromised by two other peculiarities of its ion beam compatible objective pole pieces, the upper of which is moveable laterally with respect to the lower and the magnification of which is approximately only 3X (typical high resolution objectives are 60–100X). Even with this odd collection of limitations, the resolution of the HVEM at 1 MeV in 1980 was estimated to be 0.6 nm, judged from the minimum observable size of irradiation-induced defect clusters. On a routine basis today, we estimate the resolution to be 2.0–2.5 nm, judging from the minimum observable size of Xe bubbles in Al.

For the resolution tests of the IVEM, a specimen of the usual Au islands on a thin amorphous Ge film was employed, from images of which optical diffractograms were made. The $C_s$ for the ANL IVEM objective is 2.8 mm, compared 0.9 mm for the standard side entry version of the H9000NAR. To permit ion beam incidence on the specimen at 30 degrees with respect to the electron beam, the bore of the upper pole piece is also 11 mm in diameter. The general configuration is shown roughly to scale in Fig. 2 which also shows schematically the essentials of the ion Faraday cup assembly in longitudinal section.

The ion Faraday cup assembly consists of two parts, functionally and electrically: a biased skim cup, the 2 mm exit aperture of which serves in the maintenance of ion beam flux and alignment during an irradiation and a biased Faraday cup which rotates into the ion beam path as shown in Fig. 2 for ion dosimetry. The final aperture of the ion beam is established by a 1.5 mm Ta aperture near the bottom of the upper pole piece collimator. The distance from the Faraday cup to the eucentric specimen position is about 5 cm. While there are many advantages to such a dosimetry system compared to the more common Faraday cup holder in the specimen position, there is one disadvantage which is exacerbated the lower the energy of the ions employed. In such a case, beam divergence from the Faraday cup position to the specimen may become significant, causing errors in the estimated ion dose to the specimen. We have not had sufficient experience with the Faraday cup assembly in the IVEM to establish any estimates of such error, but for the similar system in the HVEM it is estimated that the actual dose to the specimen may be 30 percent lower than that measured at the Faraday cup for the case of 50 keV Kr on $Ni_3Al$, as judged from the number density of disordered zones relative to the measured ion fluence. With a specimen such as Si, the irradiated area of which will be clearly marked, the irradiated area is larger than the final aperture size of the skim cup.

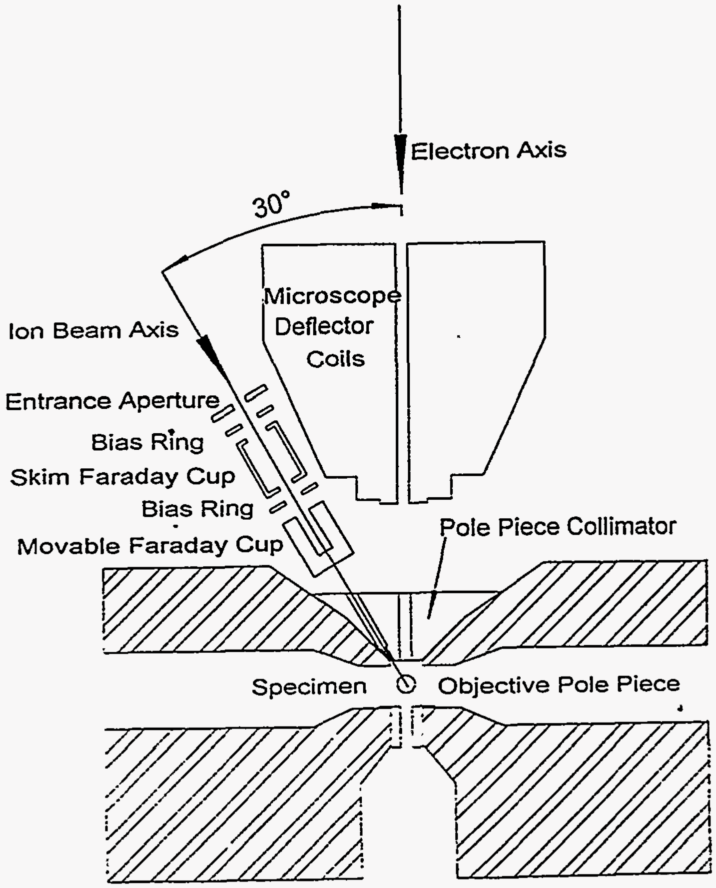

Fig. 2 Sketch of IVEM objective lens section through the ion Faraday cup assembly.

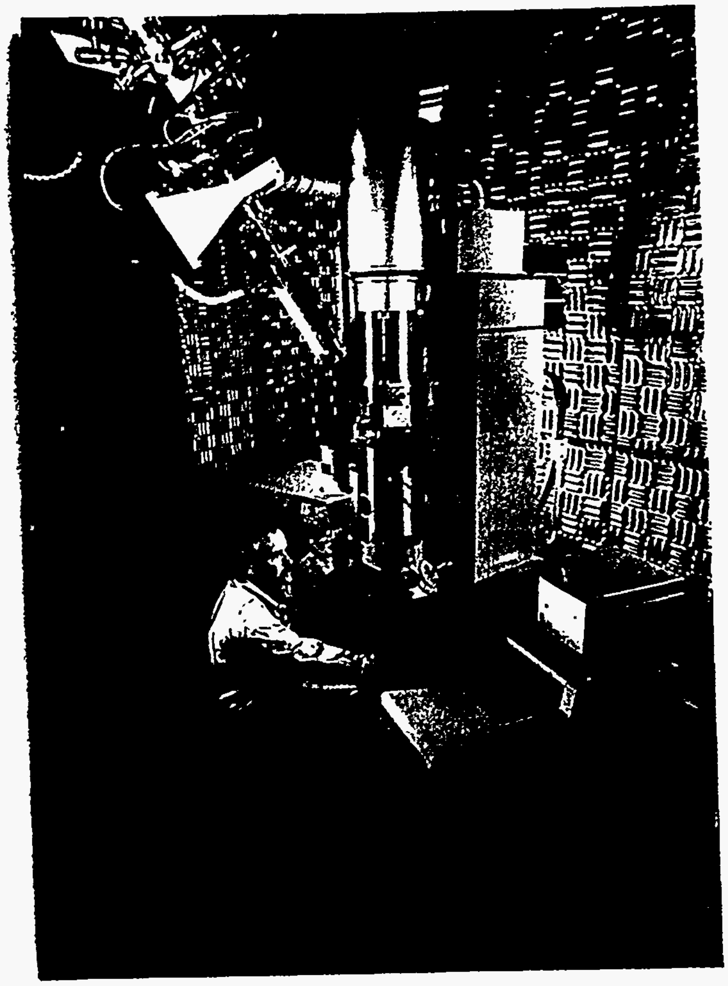

Fig. 3 Argonne Hitachi H9000 NAR and lower portion of ion beamline to which it is attached.

The photograph of Fig. 3 shows the completed installation of the IVEM and its associated ion beamline. The portion of beamline shown is suspended from two large ceiling I-beams by a triangular-shaped, tubular steel frame, the each corner of which rest on a TMC gimble piston vibration isolator. Each isolator is held in an open box which is clamped to one of the I-beams. Two very soft welded bellows units (~0.5 kg per cm of extension) connect this section of ion beamline to the IVEM and to the 60 degree electrostatic deflector of the ion beamline on the upper floor. The upper floor and the I-beams are mechanically very noisy. The attachment of the ion beamline to the IVEM, however, results in less than a 10 percent degradation of image resolution. This fortunate result is due to two factors, effective vibration isolation of the ion beam interface system and the intrinsic insensitivity of the Hitachi H9000 to mechanical inputs, the later of which was an important factor in the selection of microscope for this application.

As is clear from the ion beam specifications at the end of Table 1, there are some serious limitations at present to the maximum energies of heavier ions which can be delivered to the IVEM. The origin of these limitations is the bending magnet which is situated where the ion beamline to the HVEM (and now also the IVEM) passes through the wall shown in Fig. 1 (the magnet is not shown). The limiting field of this bending magnet places limitations on the energies of singly charged particles of mass larger than that of Ne from the tandem accelerator for which a 45 degree bend is required. The IVEM is situated in the room below the control room shown in Fig. 1.

Additional details regarding the capabilities of the HVEM-Tandem Facility and procedures for accessing its instrumentation may be obtained by writing one of the authors or by the following means: telephone: (708) 252-5222; FAX: (708) 252-4798;

e-mail: edward_ryan@qmgate.anl.gov. Request a copy of the "HVEM-Tandem Facility User Guide".

## REFERENCES

[1] D. W. Pashley and A. E. B. Presland, Phil. Mag. 6 (1961) 1003–1012.
[2] S. Ishino, J. Nucl. Mater 206 (1993) 139–155.
[3] C. W. Allen, Ultramicroscopy 56 (1994) 200–210.
[4] C. Allen, S. Ohnuki and H. Takahashi, Trans. Mat. Res. Soc. Jpn. 17 (1994) 93–96.