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Facilities for In Situ Ion Beam Studies in Transmission Electron Microscopes*

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Facilities for in situ ion beam studies in transmission electron microscopes

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Interfacing an ion accelerator to a transmission electron microscope (TEM) allows the analytical functions of TEM imaging and electron diffraction from very small regions to be employed during ion-irradiation effects studies. At present there are ten such installations in Japan, one in France and one in the USA. General specifications of facilities which are operational in 1993 are summarized, and additional facilities which are planned or being proposed are briefly described.

1. INTRODUCTION

Irradiation effects studies employing transmission electron microscopes (TEM) as analytical tools have been conducted for almost as many years as the materials community has employed TEM, motivated at least initially by materials needs for nuclear reactor development. Such studies have focussed on irradiation-induced and irradiation-enhanced microstructural and chemical changes, including phase transformations such as precipitation and dissolution, crystallization and amorphization, and order-disorder reactions. From the introduction of high voltage electron microscopes (HVEM) in the mid-1960s, studies of electron irradiation effects have constituted an important part of HVEM application in metallurgy and materials science. While electron accelerators had existed for several decades earlier, it was the introduction of HVEMs in the 1960s which made large electron flux and thus high dose studies possible in a known area of specimen with simultaneous observation and characterization of that area. For irradiation effects studies two additional developments are of especial importance, (1) the availability of specimen holders in which specimen temperature can be controlled in the range 10–2200 K during an experiment and (2) the interfacing of ion accelerators which allows in situ TEM studies of irradiation effects and the ion beam modification of materials by ion implantation.

The importance of in situ experiments in general is threefold; (1) such experiments can be performed rather quickly, often with a stream of simultaneously generated analytical information; (2) particularly for experiments at elevated or cryogenic temperatures, analysis at temperature may be essential in order to avoid mitigating effects during warmup or cooldown; (3) realtime observation of one region may be essential in correctly concluding what is the physical mechanism of a phenomenon. The purpose of this presentation is to survey briefly the present and near-term state of in situ ion beam capability internationally in the context of transmission electron microscopy.

2. FACILITIES—OPERATIONAL

At this writing there are twelve installations in the world utilizing transmission electron microscopes (TEM) with in situ ion beam capability. Table 1 summarizes several relevant specifications for each of the facilities which was operational during 1993. Also included as footnotes to Table 1 are installations which are planned to be brought into service in 1994. An excellent historical perspective of ion beam studies performed in TEM has been presented by Ishino [1].

With regard to Table 1, it is clear that the spectrum of specifications, both for TEMs and for ion accelerators is very broad. At present, three of the installations utilize high voltage

Table 1. Transmission electron microscopes with in situ ion-beam capability. Operational in 1993.

LOCATION (DATE*)	NATION	REF	ACCELERATOR VOLTAGE	TEM VOLTAGE
Argonne (1981)	USA	[2]	0.2–2 MV 20–650 kV (Not simultaneous dual ion)	0.1–1.2 MV**
CSNSM-CNRS (1980)	France	[3]	5–200 kV	100–120 kV
ERIEPI(1983)	Japan	[4]	20 kV	100–200 kV
Hitachi, Ltd. (1984)	Japan	[5]	10–400 kV	100–200 kV
Hokkaido U. (1984)	Japan	[6]	20–300 kV	0.4–1.3 MV
Inst. Phys. Chem. (1987)	Japan	-	0.4–1.0 MV	100–200 kV
JAERI—Tokai(1990)	Japan	[7]	Two 2–40 kV (Simultaneous dual ion)	100–400 kV***
JAERI—Tokai (1986)	Japan	[8]	10 kV	100 kV
Kyushu U. (1991) (Kasuga)	Japan	[9]	0.1–10 kV	40–200 kV
Kyushu U. (1988) (Hakozaki)		[10]	1–30 kV	0.25–1.25 MV
NRIM—Tsukuba (1989)	Japan	-	10–100 kV 10–30 kV (Simultaneous dual ion)	100–200 kV****
U. Tokyo—Tokai (1978)	Japan	[11]	10–400 kV	100–200 kV

* Year in which the first in situ ion beam experiment was performed.

** In 1994, 100–300 or 400 kV TEM is planned at Argonne, interfaced to existing ion accelerators. Not simultaneous dual ion.

*** Transferred from JAERI—Tokai to JAERI—Takasaki late in 1993 where IVEM is now interfaced to 40 kV and 400 kV accelerators for in situ dual ion irradiations.

**** In 1994, 0.4–1.25 MV HVEM is planned at NRIM, interfaced to 30–200 and 10–100 kV ion implanters. Simultaneous dual ion.

electron microscopes (HVEM: >400 kV) and only one involves an intermediate voltage electronmicroscope (IVEM: 300–400 kV). All but two of the sites (Argonne and CSNSM—Orsay) are located in Japan.

As indicated in the footnotes of Table 1, during 1994, an HVEM is planned for the National Research Institute for Metals (NRIM—Tsukuba) with dual ion capability, and an IVEM, for the HVEM-Tandem Facility at Argonne

Table 2. Transmission electron microscopes (TEM) with in situ ion-beam capability. Proposed.

LOCATION (DATE*)	NATION	ION ACCELERATION VOLTAGE	TEM VOLTAGE
Argonne (1996)**	USA	20–400 kV 0.3–3.0 MV (Simultaneous dual ion) and 1–20 kV 20–400 kV (Simultaneous dual ion)	0.1–2.0 MV 100–300 or 400 kV***
Hokkaido U. (1995)	Japan	20–300 kV	0.4–1.3 MV
Kyushu U. (1995)	Japan	1–50 kV 20–400 kV (Simultaneous dual ion)	0.25–1.5 MV

* Proposed starting date for project.

** Project known as HVEM-Tandem II; HVEM known as MICROLAB.

*** Intermediate voltage electron microscope indicated in second footnote of Table 1.

National Laboratory, the latter utilizing existing accelerators individually, i.e., not for in situ dual ion irradiation studies.

Most of the facilities listed in Table 1 have been developed for the research programs of specific groups within the individual institutions. The possibility for collaboration of other scientists and engineers with these groups always exists, however. In addition, the Argonne facility is funded for the materials research community specifically as a user facility, free of charge for non-proprietary studies.

3. FACILITIES—PROPOSED

Table 2 summarizes installations which have been proposed prior to the end of 1993. Each of these is an upgrade of an existing facility, which includes a new HVEM to replace aging existing equipment. Several other new installations or significant modifications of existing facilities were still in the preproposal stage in this period, and inclusion in this report was deemed to be premature therefore.

With significant improvements in microscope design and the adaptation of techniques for elemental microanalysis during in situ experiments, important scientific and technological contributions by users of these new facilities and of those already planned can be expected, which heretofore have not been possible due to experimental limitations.

Argonne National Laboratory is proposing to replace most of the equipment of the present HVEM-Tandem Facility with state-of-the-art equipment. The proposed HVEM is a 2 MV version of the 3 MV instrument constructed by Hitachi during 1992/93 for the Research Center for Ultra High Voltage Electron Microscopy at Osaka University. The specimen regions of these instruments are unusually large and accessible, designed specifically for in situ studies involving a spectrum of analytical techniques.

Hokkaido University is planning to replace the present HVEM-ion accelerator system. The new system will be employed for surface modification studies involving the implantation of various ions, including metals. In

approximately the same time frame, the HVEM Laboratory at Kyushu University is proposing to replace the present HVEM-ion accelerator system with a similar, state-of-the-art HVEM interfaced to two accelerators for dual ion studies. The new HVEM is expected to provide various functions for observation of structural, chemical and electronic changes in materials.

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- Argonne National Laboratory (Charles W. Allen) 708-252-4798; [708-252-4157 or 708-252-5222].
- Central Research Institute of Electric Power Industry (Hideo Kusanagi) 3-3480-1668; [3-3480-2111].
- Energy Research Laboratory—Hitachi Ltd. (Shigeki Oono) 294-53-2830; [294-53-3111].
- Centre de Spectrometrie Nucleaire et de Spectrometrie de Masse-CNRS (Marie-Odile Ruault) 1 69 41 52 68; [1 69 41 67 50].
- Hokkaido University (Heishichiro Takahashi) 11-757-3537; [11-716-2111 Ext. 6767].
- Institute of Physical and Chemical Research (Wako) (Eiichi Yagi) 48-462-4648; [48-462-1111 Ext. 3333 or 4671].
- Japan Atomic Energy Research Institute—Takasaki (Hiroshi Naramoto) 273-46-9687; [273-46-9420].
- Japan Atomic Energy Research Institute—Tokai (Sigemi Furuno, Kiichi Hojou) 292-82-5927; [292-82-6474 or 6360].
- Kyushu University (Hakozaki Campus) (Chiken Kinoshita, Yoshitsugo Tomokiyo) 92-641-7098, 92-632-0434; [92-641-1101 Ext. 5813 or 5970].
- Kyushu University (Kasuga Campus) (Nacaki Yoshida, Takeo Muroga) 92-582-4201; [92-573-9611 Ext. 510 or 516].
- National Research Institute for Metals (Tsukuba) (Kazuo Furuya) 298-51-4556; [298-51-6311].
- University of Tokyo (Tokai Campus) (Shiori Ishino) 33-812-1794 or 3-3818-3455; [3-3812-2111 Ext.6986].

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