HRTEM and Opportunities for Nanoscale Analysis

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Microscopy and Advanced Instrumentation Scientist

CEER Open House
October 16, 2014
Outline

• Theory of Transmission Electron Microscope (TEM)

• How CEER can support your needs
  • Research Work
  • Education
  • Service Analysis
  • Research Proposal/Collaboration

• Accessing the TEM
  • Before Using – Make A Reservation
  • Hours of Operation
  • Fees and Acknowledgment
Theory of TEM
Theory of TEM

Theory of TEM

Electron gun

Layout of a basic TEM

Theory of TEM

Theory of TEM

How CEER can support your needs
How CEER can support your needs

Research Work

- Key Product Features of JEM 2100F system
  - High Resolution TEM (HRTEM) equipped with Field Emission Gun
  - Resolution: 0.1 nm (lattice image) / 0.19 nm (point image)
  - Accelerating Voltage: 200 kV
  - Magnification: \( \times 2,000 - 1,500,000 \)
  - Specimen Stage: Micro active goniometer
  - Specimen Chamber
    - Specimen per load: 1
    - Specimen Tilt Angle (X-axis): \( \pm 35^\circ \)
    - Specimen Tilt Angle (Y-axis): \( \pm 30^\circ \)
How CEER can support your needs

Research Work

- Charge-Coupled Device (CCD) Camera
- Energy-Dispersive X-ray Spectroscopy (EDS) detector\(^2\)
- Scanning TEM (STEM) detector
- Single-tilt sample holder
- Double-tilt sample holder

How CEER can support your needs

Research Work

• Poseidon electrochemistry liquid cell holder[3]

How CEER can support your needs

Research Work

• TEM imaging at nanoscale
  • HRTEM
  • Bright Field Imaging
  • Dark Field Imaging
  • STEM
  • 3D Tomography

How CEER can support your needs

Research Work

- TEM imaging at nanoscale
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TEM and HRTEM images at typical evolution stages from polyhedra to nanoframes\textsuperscript{[5]}

\textsuperscript{[4]} Nano Lett. 12(9), 4417 (2012)
\textsuperscript{[5]} Science 343, 1339 (2014)
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Bright field and dark field STEM image of PtNi\textsubscript{3} nanoframes\textsuperscript{[5]}

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TEM and HRTEM images at typical evolution stages from polyhedra to nanoframes

Bright field and dark field STEM image of PtNi₃ nanoframes

3D reconstruction of nanocatalysts

References:
How CEER can support your needs

Research Work

• Crystal structure analysis
  • Selected Area Electron diffraction (SAED)
  • HRTEM

SAED pattern, HRTEM and growth scheme of Pt planar tripod[7]

HRTEM images of Ag nanocrystals and nanoplates[6]

How CEER can support your needs

Research Work

• Elemental composition analysis
  • Energy-Dispersive X-ray Spectroscopy
  • Chemical Elemental Mapping
  • EDS line scan

How CEER can support your needs

Research Work

- Elemental composition analysis
  - Energy-Dispersive X-ray Spectroscopy
  - Chemical Elemental Mapping
  - EDS line scan

Elemental mapping of PtNi₃ polyhedron and nanoframe[^5]

[^5]: Science 343, 1339 (2014)

Combinations of elemental maps of Se and S in selected nanocrystals[^8]
How CEER can support your needs

Research Work

- Elemental composition analysis
  - Energy-Dispersive X-ray Spectroscopy
  - Chemical Elemental Mapping
  - EDS line scan

STEM images and EDS line scans along the axis of individual nanocrystals\(^8\)

Elemental mapping of PtNi\(_3\) polyhedron and nanoframe\(^5\)

Combinations of elemental maps of Se and S in selected nanocrystals\(^8\)

\[\text{[5] Science 343, 1339 (2014)}\]
How CEER can support your needs

Research Work

- *In-situ* study in liquid environment
  - *Real-time electrochemistry process*
  - Biological application

Time evolution of the growth and dissolution of Li-Au alloy and lithium dendrite

A scheme of *in-situ* TEM observation using an electrochemical liquid cell

How CEER can support your needs

Research Work

- *In-situ* study of reactions
  - Real-time electrochemistry process
  - *Biological application*

Light microscopy and liquid STEM of fully hydrated wild-type S. pombe yeast cells, which were alive at the onset of the recording of the first STEM image[^10]

[^10]: Biophysical Journal 100, 2522 (2011)
How CEER can support your needs

Education

• Training to be certified as TEM users
  – Graduate students, Postdocs, Scientists and Professors
  – Professional training program (1 person at a time)
  – At least Three TEM sessions (4 hours each) are required
  • 1st session*: introduction/demonstration of TEM provided by CEER Microscopy Scientist
  • 2nd session: operation of TEM with assistance
  • 3rd session: operation of TEM independently and evaluation by CEER Microscopy Scientist
  – Certification will be granted after passing the training program, which can be added to user’s resume
  – User is authorized to operate JEM 2100F independently

*Bring well-prepared samples ready for TEM observation and confirm with Microscopy Scientist or inform Microscopy Scientist in advance to use sample provided by CEER
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How CEER can support your needs

Education

• Safety during operation of the TEM
  – Capabilities of the TEM
  – Parts and components of the TEM

• How to obtain high quality TEM images
  – Sample preparation
  – Basic TEM alignment
  – Record digital high quality TEM images

• Instrumental Assistance of Advanced Techniques
  – EDS, Elemental Mapping, STEM
  – User is recommended to present during the measurement
How CEER can support your needs

Education

• Class support
  – Short lecture upon request
  – For undergraduates, graduates or even high school students
  – Introduction of TEM basic theory and principles
  – Capability and applications of TEM
  – Remote demonstration of TEM operation (Sirius Remote Software)
  – Tour of TEM lab (5 people/group maximum)
  – Not equivalent to TEM training program
  – Contact CEER for details and reservations
How CEER can support your needs

Service Analysis

• Send in the samples, we provide professional services
  – TEM, HRTEM images
  – SAED patterns, EDS spectra, Elemental Analysis

• Consultation before and after measurement*
  – Expect significant communication
  – Required consultation before making reservation
  – How to best fit your needs
  – Saves trouble and time

• Deliver the original data (CD) and analysis report* to customer

• Retrieve or disposal of the samples at customer’s expense

* Upon request, may add additional charges
How CEER can support your needs

Research Proposal/Collaboration

• Strong Research Team
  – Dr. Yuxuan Wang, Microscopy and Advanced Instrumentation Scientist
  – Dr. Gerardine Botte, Russ Professor/Director of CEER

• Extensive Experience
  – High quality TEM, HRTEM data acquisition
  – SAED, EDS data interpretation
  – Electrochemical Engineering background
  – Nanoscience knowledge

• Advanced Equipment
  – JEM 2100F Field Emission HRTEM
  – XRD, AFM, FTIR, Raman
  – Electrochemistry station
  – Wet-chemical research lab
Accessing the TEM
Accessing the TEM

Before using the TEM – Make a reservation

- Complete required safety training
  - Chemical Hygiene Training
- Sign necessary forms
  - Internal Use Agreement Form
  - Radiation Acknowledgment Form
  - Training Agreement form (to be certified user)
Accessing the TEM

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Accessing the TEM

Before using the TEM
- Make a reservation
- Schedule the user's appointment

User's Agreement

I, (User), agree to use the following instrument in accordance with the

Instrument Name: TEM 2100F

Attachment: None

Usage Rate: $10.00 per hour (as operated by user)

Usage Rate: $40.00 per hour (as operated by CEER Technician)

Data收集: Contact CEER Technician at 740.593.1500

Cancellations: User appointments must be cancelled through Facility Office Manager (FOM) online within 24 hours prior to the appointment. If the notice is not received within 24 hours prior to the appointment, a fee equal to one hour of usage will be charged to the account listed.

No Show Policy: If the user doesn’t keep the appointment, a fee equal to half of the estimated usage cost, or the cost of one hour of usage when the reserved time is less than two hours, will be charged to the account listed.

Service Requested: Machine operated by User for an estimate of __ hours

Machine operated for CEER Technician for an estimate of __ hours

Data collected: (including original data, experimental conditions and expected results)

Additional setup and parts may be required and may add an additional hour(s) of usage.

CEER Microscopy and Advanced Instrumentation Scientist have discussed the User’s equipment needs and verified the estimates of hours listed above.

Microscopy and Advanced Instrumentation Scientist’s Signature ____________________________

Final Total Amount Charged may deviate from above estimate and will be provided to Administrative Budget Controller.

Are the measurements going to be used in a publication? __ Yes __ No

Publication Journal ____________________

Date of submission ____________________

CEER will be acknowledged in the resulting publication/presentation. The acknowledgment should read:

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Ohio University

Center for Electrochemical Engineering Research
RUSS COLLEGE OF ENGINEERING AND TECHNOLOGY
Accessing the TEM

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Accessing the TEM

1. Before using the TEM
   - Make a reservation

2. In compliance with Rule 3701-10-03 of The Ohio Department Health Radiologic Technology section, individuals must be informed of the proper use of radiation-generating equipment as well as any health risks and safety precautions.

3. What is the restricted area and where is it located?
   - A restricted area is any area where radiation is produced. The TEM room of the CER analytical lab is designated a restricted area.

4. Have the restricted area been posted and - as specified in your "Standard Operating Procedure" - posted outside the door of each TEM room of CER analytical lab in radiation containment that states, "STAFF AUTHORIZED PERSONNEL ONLY". Access to the TEM area is restricted to individuals who have been authorized by CER personnel and by Dr. Bernarda Cesar, Director of CER.

5. What is the radiation source and where is it located?
   - The JEM-2100F TEM system is located in the CER analytical lab.

6. What are the biological effects of radiation exposure?
   - Biological effects of radiation vary in nature, age, and cause that are harmful to health due to radiation exposure.

7. How can occupational exposure to be kept at a Low As Reasonably Achievable (LARA) at this facility?
   - Disposal of radiation can be controlled or monitored with the use of lead shielding, proper techniques, distance and time. Since the TEM is self-shielding equipment, there should be no exposure to radiation while operating it.

8. What radiation protection policies have been created specifically for users of this facility?
   - The radiation protection policies can be found in "Standard Operating Procedure for users of Transmission Electron Microscopy JEM-2100F".

9. Where are the "Standard Operating Procedures"?
   - The "Standard Operating Procedure for Users of Transmission Electron Microscopy JEM-2100F" is located on the right side of the TEM left control panel.

10. Where is the Ohio Department of Health's "Notice to Employee" posted?
     - The Ohio Department of Health's "Notice to Employee" is posted on the door of TEM room, CER analytical lab.

Who to contact for inquiries or concerns on radiation protection:
For inquiries or concerns on radiation protection, please contact Alex Wirtz (Ohio University Radiation Safety Officer/Health Physicist).

What reports of radiation exposure are available and where are they kept?
Annual inspection reports, which include radiation exposure measurements, are kept at the Radiation Safety Office, Risk Management and Safety, 1179 University Service Center.

What is the level of radiation exposure users can receive?
No user shall exceed an exposure of 5 rem per year for whole body and 1.58 rem per quarter-year.

As a user, have reviewed and understood the information in the Radiation Safety Acknowledgment provided by the trainer of CER.

User Name (print): ___________________________ Signature: __________ Date: _______

Trainer Name (print): ___________________________ Signature: __________ Date: _______
Accessing the TEM

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Accessing the TEM

Before Using the TEM – Make A Reservation

- Register online as Facility Online Manager (FOM) user
- Choose the accessible instrument after login
- Check availability on the FOM calendar
- Click future time on the calendar to make a reservation
- Reserve 48 hours in advance, at least
- Contact CEER personnel if special service is required
- CEER personnel has the right to make necessary changes

http://ceer.dynohio.net/fom/
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Accessing the TEM

Before Using the TEM – Make A Reservation

User homepage: Authorized users have access to available instruments, service and collaboration records, documents, and reports of their own usage. Users may sign on instruments from multiple facilities with single sign on.

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Accessing the TEM

Before Using the TEM – Make A Reservation
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http://ceer.dynohio.net/fom/
Accessing the TEM

Before Using the TEM – Make A Reservation

Instrument reservation: Administrators may reserve instrument for various purposes. Admin can also reserve session for another user.

Facility Online Manager - Schedule

Before Using the TEM

– Make A Reservation

 instrument
 manager

Make A Reservation

Select the purpose of this reservation:

- For repair
- For training
- For paid service research
- My own research
- Reserve for a user

Start time: 2009-04-05 21:00:00
End time: 2009-04-05 21:30:00

Reserve Cancel
Accessing the TEM

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http://ceer.dynohio.net/fom/
Accessing the TEM

Hours of Operation

• Day-time access
  – 8:00 AM - 5:00 PM
  – Monday – Friday, excluding holidays

• Recommended reservation slots
  – Reserve a 4 hour slot
  – Usually, at least 30 min/sample for TEM imaging
  – 8:00 AM – 12:00 PM & 1:00 PM – 5:00 PM
Accessing the TEM

Fees

- **Internal Rates**
  - CEER Staff, $140 / hour
  - User, $105 / hour
  - Much cheaper, more convenient
  - Rates may change in future
  - Rates were calculated based on maintenance/service cost and total yearly TEM usage and approved by the Grants and Contract Accounting Office of Ohio University

- **Cancellation Policy**
  - 24 hours in advance, no charge
  - Within 24 hours, 1 hour of usage cost will be charged
  - No show, ½ of total reserved usage cost will be charged, with a minimum of $140

<table>
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<th></th>
<th>OSU</th>
<th>Miami U</th>
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</tr>
<tr>
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</tr>
</tbody>
</table>
Accessing the TEM

Acknowledgment

- Acknowledgment
  - Are the measurements going to be used in a publication? If yes indicate:
    - Publication journal
    - Tentative date of submission
    - Inform CEER about the publication (journal, pages, title, etc)
  - Acknowledgments for publications and presentations

“We would like to thank the Center for Electrochemical Engineering Research (CEER) at Ohio University, and the National Science Foundation through the Major Research Instrumentation Grant # CBET-1126350 for the Transmission Electron Microscopy images and measurements.”
Thank you

Questions: CEERfacilities@ohio.edu
http://www.ohio.edu/engineering/ceer/