

Installation of JEOL ARM-200CF

Technical Summary NSF Award (15th March 2010)

MRI-R2: Acquisition of an Aberration-Corrected Scanning Transmission Electron Microscope for Multidisciplinary Research and Education at UIC

This award is funded under the American Recovery and Reinvestment Act of 2009

Transmission electron microscopy (TEM) is an essential tool in the study of atomic-scale structure-property relationships of functional nano-materials. With the development of aberration correctors, the spatial resolution of TEMs operated at 200kV has now reached 70 pm. While most aberration-corrected TEMs are optimized to achieve the highest spatial resolution, their analytical capabilities are limited by a moderate energy-resolution (~0.8 eV at 200 kV) and the inability to perform in-situ experiments. This project will acquire a next-generation aberration-corrected TEM, with unprecedented spatial and energy resolution for multidisciplinary research and education at UIC. The JEOL ARM 200CF incorporates many recent innovations including a next-generation spherical-aberration probe-corrector (70 nm spatial resolution), a high-brightness cold-field emission electron source (0.3 eV energy-resolution), variable acceleration energy (between 80-200 kV), as well as improved mechanical, thermal and magnetic shielding. The instrument is unique in its abilities to provide ultra-high resolution for imaging and spectroscopy in the temperature range between 80-1000 K using in-situ heating/cooling holders. It provides the faculty involved in this project the unique abilities to study a broad range of interdisciplinary problems, including correlated behavior in complex oxides, thermal stability in metal/oxide interfaces, heat transfer filled carbon nanotubes, as well as the interaction of airborne nanoparticles with human cells. The instrument will be operated by UIC's Research Resources Center, a center of excellence for teaching and training of advanced TEM. The faculty at UIC will further use the instrument to develop strong collaborative interactions with groups at surrounding academic institutions and industrial laboratories to maximizing the outreach opportunities for training and research of UIC's diverse student body.

UIC Press Release (30th March 2010)

University of Illinois at Chicago Office of Public Affairs (MC 288) 601 S. Morgan St., Chicago, IL 60607-7113, (312) 996-3456,

New Electron Microscope Promises Sharper Nano-Viewing

The University of Illinois at Chicago will become the first university in the world to have a new generation of electron microscope, promising views up to three times sharper than instruments now commonly used and providing a unique tool for the Midwest's academic and industrial research community.

Robert Klie, assistant professor of physics, was awarded a \$2 million National Science Foundation MRI² grant to acquire an aberration-corrected scanning transmission electron microscope, or STEM. The sophisticated instrument will let scientists see individual atoms, helping them better understand how materials function.

"By improving the resolution we can decrease the blurring, so we'll see atoms that were previously indistinguishable," said Klie, who previously worked with a comparable instrument at the Brookhaven National Laboratory.

Unlike optical microscopes that use visible light to illuminate a sample, transmission electron microscopes view samples using a carefully controlled beam of electrons accelerated to nearly the speed of light. With aberration correction, UIC's STEM will provide sharper images and have reduced electron energy blur, which improves color distinction.

"UIC is the first of any university in the world to get a STEM with these specific capabilities," Klie said. "It's unique."

UIC's STEM will also be the only one in the Midwest, and one of only a few in the U.S. Comparable instruments are at the Brookhaven, Oak Ridge and Lawrence Berkeley national laboratories.



UIC's STEM will allow a range of electron velocities. Slower electrons will limit damage to biological samples and certain nanostructures such as nanotubes, while faster electrons will enhance and sharpen images through the state-of-the-art aberration correction feature.

"With the aberration correctors, you can correct for the decreased resolution of slower electrons and still get the atomic resolution, even at these low acceleration voltages," Klie said.

The new instrument will mark the first significant upgrade to UIC's electron microscopy facility in over a decade.

Klie said it will offer students unparalleled training opportunities, providing unique skills for such in-demand job fields as green energy, electronics, petrochemicals and pharmaceuticals.

He adds that it will not only greatly benefit interdisciplinary research at UIC, but will complement Chicago-area electron microscopy facilities at Argonne National Laboratory and Northwestern University.

Installation of the aberration-corrected STEM will be completed within a year. It will be housed with two conventional transmission electron microscopes at the UIC Research Resources Center's Science and Engineering South facility.

UIC will contribute 30 percent to the cost of the NSF-funded project, made possible by the American Recovery and Reinvestment Act of 2009.

Klie's co-principal investigators are Sivalingam Sivanathan, physics; Neil Sturchio, earth and environmental sciences; Luke Hanley, chemistry; Christos Takoudis, chemical and bioengineering; G. Ali Mansoori, bioengineering; Randall Meyer, chemical engineering; David He, Farzad Mashayek and Constantine Megaridis, mechanical and industrial engineering; Siddhartha Ghosh, electrical and computer engineering; Serap Erdal, environmental and occupational health sciences; and Alan Nicholls, electron microscopy, Research Resources Center.

For more information about UIC, visit www.uic.edu

Order placed with JEOL for JEM-ARM200CF (13th October 2010)



JEOL JEM-ARM200F Probe Aberration Corrected STEM/TEM with Schottky Field Emitter (Note microscope UIC is buying will have a cold field emitter (ARM-200CF))

A purchase order was placed with JEOL USA on 13th October 2010 for the **JEOL JEM-ARM200CF** to be delivered in 2011.

The new microscope will have:-

- a **cold field emission** source giving better than 0.4eV resolution.
- a **probe spherical aberration corrector** for STEM imaging.
- a **HADF-STEM imaging resolution** of better than **78pm** with a target resolution of 70pm (TEM phase contrast resolution will be 140pm).
- stage positioning will be in 0.1nm increments with a drift rate of 0.05nm/min.
- a **Gatan Enfina 1000 EELS** system with high speed detector capable of readouts of >200 spectra/sec with STEM spectrum imaging capability.

- an **Oxford Inca Energy TEM 250 XEDS** system with an 80mm² **X-Max 80** Silicon Drift Detector with a solid angle greater than 0.2srad and energy resolution of better than 129eV at Mn K. It will also have STEM quantitative spectrum imaging capability.
- two retractable CCD cameras for TEM imaging and diffraction. One mounted above the viewing screen will be a **Gatan Orius SC200D** 2kx2k side entry lens coupled camera for high resolution imaging and streak free diffraction patterns with a readout of up to 30fps. The second mounted above the EELS will be a **Gatan Orius SC1000B** 11MP fiber optic coupled camera capable of up to 14fps.
- Two HAADF detectors, a LAAADF detector, a BF detector and an ABF (annular bright field detector) for STEM imaging. One of the HAADF detectors will be close to the EELS spectrometer to allow simultaneous HAADF imaging and EELS spectroscopy.
- Heating, Cooling and Tomography stages.

In addition the ARM series has been designed to minimize environmental factors

- Environmental measures - The JEM-ARM200F offers the ultimate stability for imaging and analysis at the atomic scale. The electron column is isolated from the environmental disturbances found in most labs. A superior shielding design safeguards the ultrahigh-powered optics from airflow, vibration, acoustical, and electronic interference, magnetic fields, and thermal fluctuations.
- Enhanced mechanical stability - To enable atomic level analysis and imaging - utilizing spherical aberration correctors for electron probe forming systems - vibration and distortion on the atomic level need to be controlled. The ARM 200F features an overall mechanical strength twice higher than conventional TEMs. Enlarging the column size improves rigidity and optimizing the console structure enhances mechanical stability.
- Enhanced electrical stability - The ARM200F reduces the fluctuation of the high voltage system and the objective lens current by 50% of that of conventional TEMs, significantly enhancing its electrical stability.

Update on delivery of the JEOL JEM-ARM200CF



< The UIC JEOL JEM-ARM200CF is seen during testing in the JEOL factory in Japan, at the end of January 2011

The microscope was shipped in March 2011 (it was on a boat in Tokyo harbor when the Tōhoku earthquake and tsunami struck on 11th March).

Delivery of the new JEM-ARM200CF is scheduled for **11th July 2011**. Modification work on Rooms 104A and 104B for the installation of the new microscope will start during June 2011.

JEOL JEM-2010F Removal



The first stage of the ARM installation - removal of the JEM-2010F - began at 9.30am on Monday, 6th June 2011 when the field emission source was switched off for the last time at UIC. During the day the gun chamber, X-ray detector and Gatan Imaging Filter were removed from the column.

< The JEM-2010F at the end of the first day of preparation for removal, Monday 6th June, 2011.

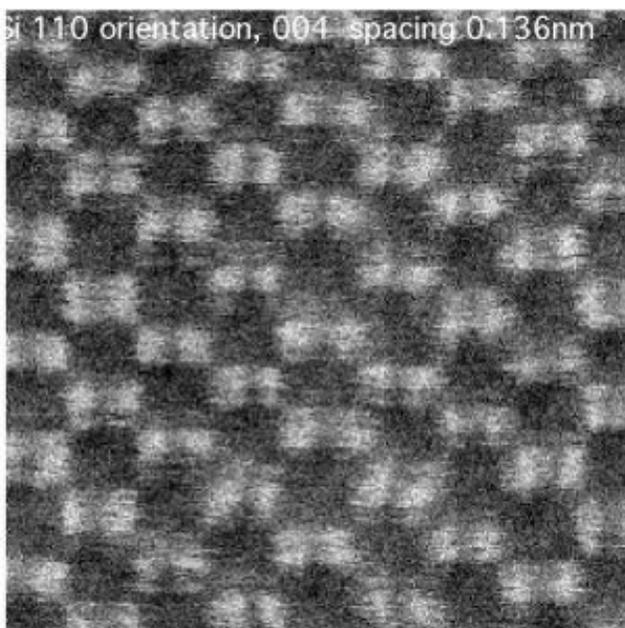
Complete disassembly of the microscope took four days.

The JEM-2010F column being removed from Room 104A >

The packers and shippers arrived early on Friday 10th June and the column, which was the last part of the microscope to be moved, was removed from Room 104A at 1:55pm.

Installation of the JEM-2010F started on 12th January 1998 so the JEM-2010F has been at UIC for nearly 13.5 years. During that time we have had two Schotky field emission electron sources. The first, replaced in November 2007, had 80,301 hours of use and the second 29,575 hours.

The UIC JEM-2010F was the first 200kV TEM/STEM to image {004} atomic columns in Si <110> by high angle dark field STEM atomic resolution imaging, seen in image below taken on December 24th, 1998. With the arrival of the JEOL-ARM200CF UIC will again be at the forefront of materials science microscopy.



< Z contrast image of Si <110> showing resolution of 0.136nm {004} planes

JEOL JEM-ARM200CF delivery

The new microscope arrived, on schedule, on Monday, 11th July 2011



The microscope arrives - 20 crates and pallets in a 40' truck.



The first part of the microscope to be moved to RRC was the HT tank



Robert Klie stands next to the uncrated lower column of the ARM200CF



The ARM200CF column is brought carefully into RRC-E



The lower column has to be accurately placed between the beams in Room 104A



*The ARM200CF lower column
in its final position*



*Most contents of the crates and pallets in
Room 109 SES ...*



... and in the main corridor of RRC-E!

JEOL JEM-ARM200CF installation (12th July – 2nd December 2011)

Installation of the microscope started Tuesday, 12th July, 2011, the day after delivery. Installation tests were completed on 2nd December and Robert Klie accepted the instrument for use by UIC on 8th December.



*July 12th. JEOL engineer adjusting one of the four
temperature sensors which controls the Hydro Radiant
Panel system used to maintain the tight temperature control
needed for the ARM200CF*



*July 14th. Having cabled the column and electronics the
system is switched on for the first time.*



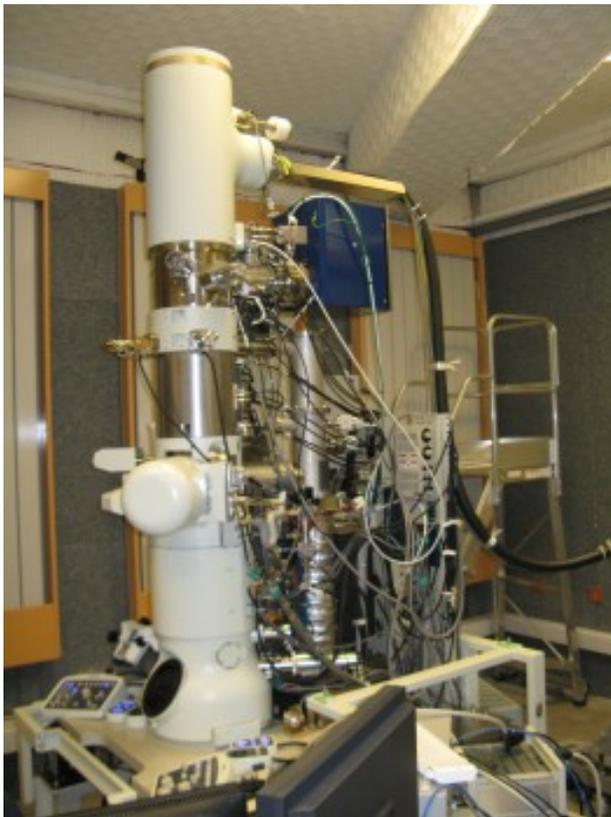
July 18th. Column after installation of condenser and corrector stack.



July 19th. Lifting the cold field emission gun onto the column.



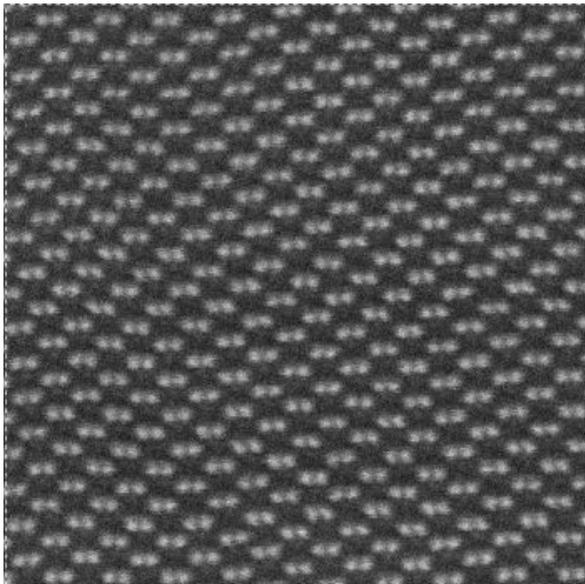
View of column after installation of cold field emission gun.



< 5th August. After checking out the HT and gun the column was baked for a week before testing starts.



Control Electronics in adjacent room (104B).



11th August. First HADF STEM images of Si $\langle 110 \rangle$ obtained, showing resolution better than the JEM-2010F, without tuning the corrector.



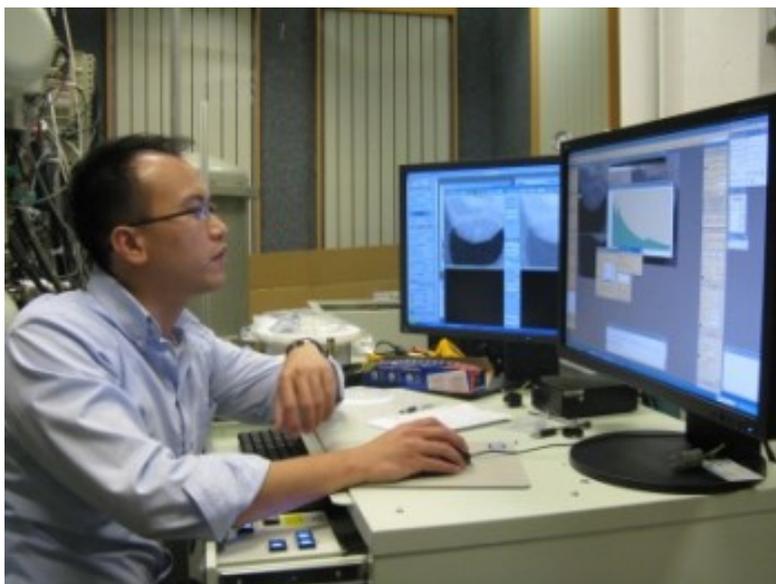
15th-18th August. First Gatan visit to install SC200D and SC1000 CCD cameras along with a fourth STEM detector and Digiscan.



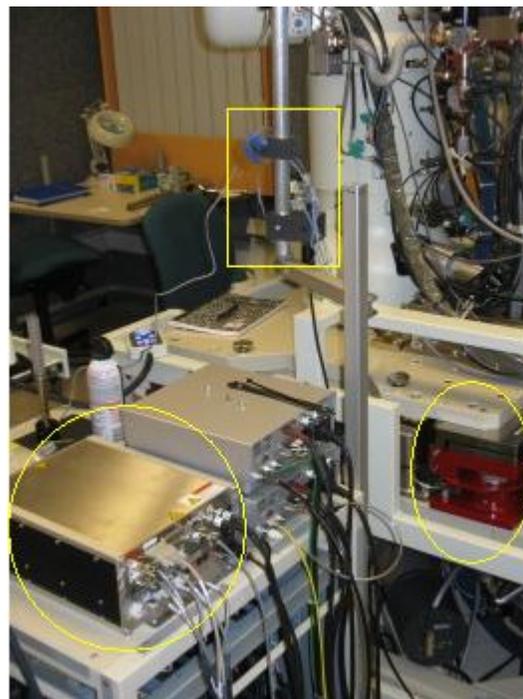
14th September. Gatan Enfina EELS spectrometer installed on the microscope.



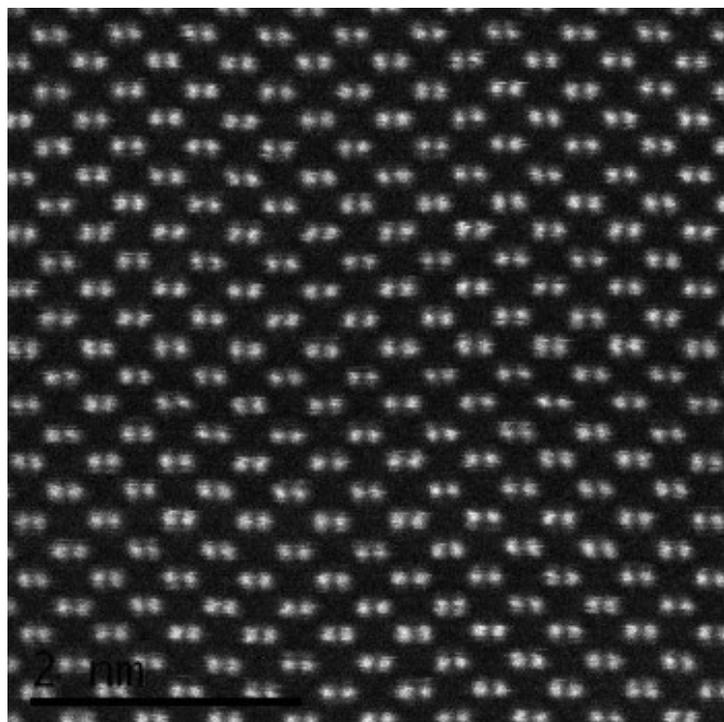
21st September. ARM200CF with lower covers in place enclosing the Enfina and detectors



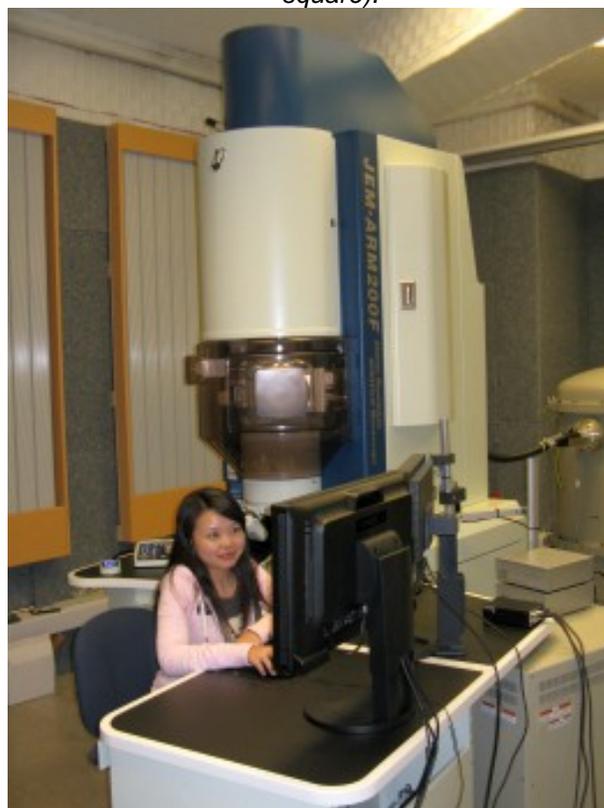
22nd September. Ke-Bin gets trained on the new Digital Micrograph EELS software.



4th-6th October. Installation of IDE active antivibration system (control box and one foot in circles) and EMF cancellation system (sensors in square).



6th October. After installation of the IDE systems the Si<110> images show sub 0.1nm resolution. This is still without the covers on the column.



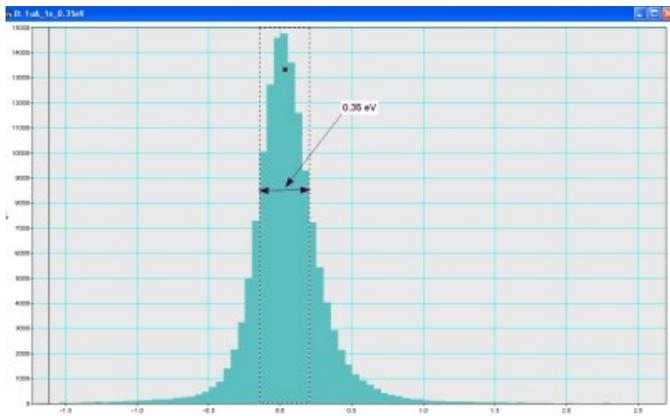
13th October 2011. ARM200CF after column covers were fitted



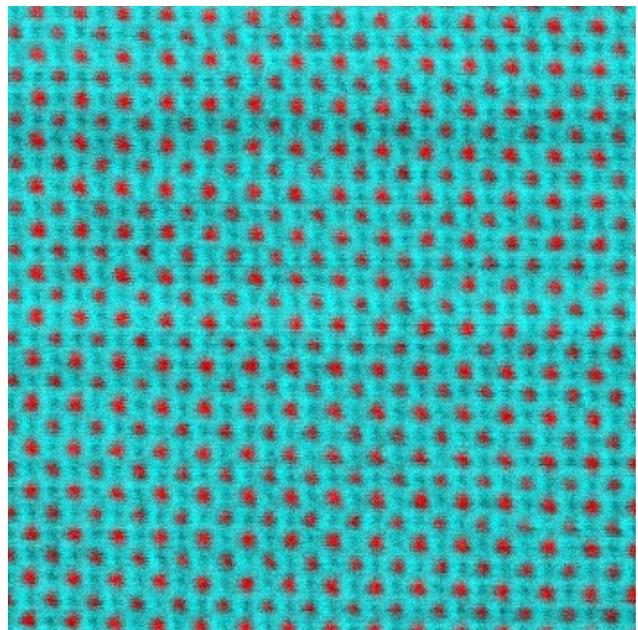
21st October. The SDD detector and column vacuum adaptors.



21st October 2011. SDD detector installed on column.



9th November. 0.35eV energy resolution demonstrated.



7th December. Composite HADF and ABF images of SrTiO3 obtained during training. (Red - HADF showing location of Sr and Ti, Blue - ABF showing location of O)

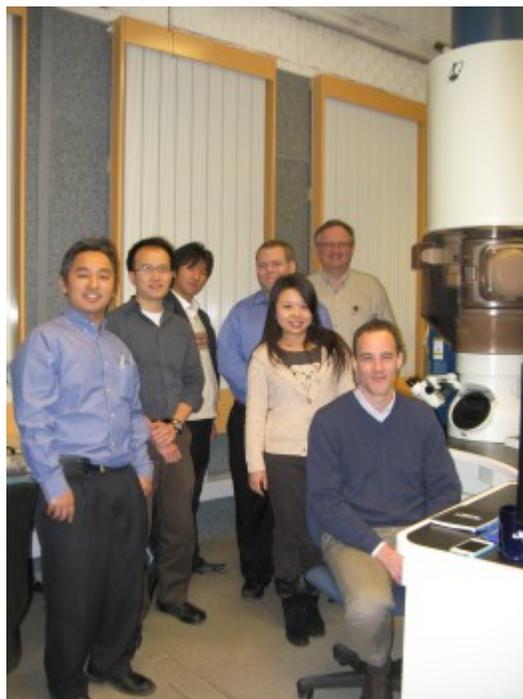
JEOL JEM-ARM200CF Certificate of Installation Signed (8th Dec 2011)

On the 8th December 2011, following consideration of the *JEOL Performance Report* by Ke-Bin Low, Alan Nicholls and Robert Klie, the Certificate of Installation was signed allowing UIC to start using the instrument. Final sign off will follow, during the first quarter of 2012, once outstanding accessory items have been received.

Initial training of RRC-EMS staff and some members of Robert's Nanoscale Physics Group took place from 5th to 9th December.



JEOL engineer Micky Woods and UIC's Robert Klie at the sign off.



Group photo at the ARM200CF. Robert Klie sitting, standing behind him from left to right are Toshi Aoki (JEOL), Ke-Bin Low (UIC), Tetsuhiko Suga (JEOL), Micky Woods (JEOL), Qiao Qiao (UIC) and Alan Nicholls (UIC).

JEOL JEM-ARM200CF Dedication Ceremony (20th January 2012)

A dedication ceremony and ribbon cutting was held on 20th January 2012 to mark the opening of the JEM-ARM200CF to researchers. Speeches were given by Robert Klie (Physics), Astrida Tantillo (interim Dean, LAS), Peter Nelson (Dean, Engineering), Joe "Skip" Garcia (Vice Chancellor for Research) and David Hoffman (Acting Head, Physics). This was followed by the ribbon cutting, and an opportunity for guests to view the new microscope.

For more information about the ceremony please see the [LAS Newsletter \(AtLAS\)](#) articles.



Guests gather to listen to the speakers at the dedication ceremony for the JEOL JEM-ARM200CF.



Joe "Skip" Garcia (Vice Chancellor for Research) speaks at the dedication ceremony.



Robert Klie (Physics), Astrida Tantillo (interim Dean, LAS), Peter Nelson (Dean, Engineering), Joe "Skip" Garcia (Vice Chancellor for Research) prepare to cut the ribbon opening the JEOL JEM-ARM200CF



Ke-Bin Low (RRC), Robert Klie (Physics), Tom Isabell (JEOL) and Alan Nicholls (RRC) in the microscope lab



Robert Klie describes how the microscope works to Deans Nelson and Tantillo.



Robert Klie (Physics) with graduate student Qiao Qiao (Physics) in front of the JEOL JEM-ARM200CF

M3S meeting: *Seeing is Believing - Aberration Corrected STEM*

On 26th April 2012 the Nanoscale Physics Group and Electron Microscopy Service hosted a one day meeting for the Midwest Microscopy & Microanalysis Society (M3S) on the topic of "**Seeing is Believing - Aberration Corrected STEM**". This gave the RRC an opportunity to introduce our new microscope to the wider Midwest community. The meeting, which was financially supported by LAS, OVCR and the Department of Physics from UIC as well as JEOL, Gatan and Oxford Instruments, was held in Student Center East, followed by an afternoon reception held in RRC-E with JEOL providing the refreshments.

Speakers including Steve Pennycook (ORNL), Nigel Browning (PNNL), Paul Voyles (UW-Madison), Yan Xin (FSU), Miguel Yacaman (UT-San Antonio), Steven Mick (Protochips) and Masashi Watanabe (Lehigh) gave a broad insight to the fields of aberration corrected STEM and in-situ microscopy to nearly 100 attendees. Patrick Phillips (UIC) introduced the UIC JEOL JEM-ARM200CF before participants moved to RRC-E to see the new microscope.



Christos Takoudis and Tolou Shokuhfar talk to the first two speakers Steve Pennycook (2nd left) and Nigel Browning (3rd right), while Robert Klie and Patrick Phillips help Yan Xin set up for her presentation.



Neil Rowlands (Oxford) introduces AZtec to the M3S members.



Alan Nicholls describes the ARM to a group of meeting participants



Everyone enjoyed the reception, sponsored by JEOL