

Final Report Solar DOE

DOE award number DE-FG36-06GO86073

Name of recipient: Oregon Nanoscience and Microtechnologies Institute

Project Title: “Oregon Nanoscience and Microtechnologies Institute”

Identification Number: DOE/GO/86073-1

Name of project director/PI: Robert “Skip” Rung

Authorized distribution limitations

No distribution limitations

1. Executive summary

To achieve its goals in meeting future clean energy requirements, the United States must develop well trained people, and the steady stream of scientific and technical innovations they produce. Education in the emerging fields of nanoscience is expected to be critical in this endeavor. Access to the basic tools used in understanding nanoscience is lacking in the education environment. The goal of this program was to develop affordable electron microscopes for nanotechnology undergraduate education, student research experiences, and workforce training. The outcome was to complete the development and delivery of tools to education institutions for evaluation. The evaluation of the tools was accomplished under a second DOE funded effort, DE-FG02-06ER64248 “Tools for Nanotechnology Education Development”, and administered by the Biological and Environmental Research (BER) division. The final report from that program is attached to this report as an appendix as a courtesy.

2. Comparison of the actual accomplishments with goals and objectives of project

The project objectives were to develop a table top scanning electron microscope (SEM) that has the following key attributes:

- a. Easy to use by non-experts and students
- b. Requires no special facilities
- c. Affordable by postsecondary educational institutions

The expected outcome was to complete the development and deliver tools to education institutions for evaluation.

The program tasks were to:

- a. Develop aspects of a SEM suitable for use within a post secondary education environment by developing aspects of recent research in electron microscopy. The critical areas of focus and development included reliability, ease of use, and design for affordability.
- b. Develop a simulator for classroom use in conjunction with the SEM. The approach was creation of software code to fulfill specifications. The outcome was a reliable software simulator able to run on common platforms found in universities.
- c. Deliver and support 6 prototype systems to colleges and universities for evaluation. The purpose was to provide SEMs for educational evaluation. The approach was to build and test six prototype tools and prepare them for delivery. The outcome was one tool delivered to each site. Each tool was fully functional and capable of being used and evaluated in an educational environment.
- d. Program management – The principal investigator and subcontractors provided overall supervision of the program, via program reviews, discussions with university professors, reports and presentations, and site visits.

3. Actual accomplishments

All of the goals and objectives of this program were met.

The education evaluation of the SEM by the universities was done under a separately funded effort (**BER Department of Energy Grant No. DE-FG02-06ER64248 “Tools for Nanotechnology Education Development”**) and is provided as an appendix to this report as a courtesy.

The key tasks are highlighted and summarized in this section. Detailed reports of each of these tasks are available, but are summarized in this final report.

Task 1 Develop aspects of a scanning electron microscope to make a SEM that is applicable to education and student environment.

The key elements of this task included improving reliability, ease of use and design for affordability.

The ease of use subsystem improvements included improving the design of certain electronics boards, software and graphical user interface (GUI) by minimizing the number of variable functions to the user, thus making the final tool easier to use.

The cost reduction effort was focused on reducing the cost of some of the most expensive elements while ensuring that the final design is consistent with performance requirements. Three particular tool elements were redesigned: the sample holder and associated injection molds for manufacturing, the source module, and a lower cost detector with associated tooling and process.

The final tool which was delivered to each of the participants embodies the improvements described in the project reports, and is shown below in the following figures.

Figure 1 Image of the Final system Design of the Table Top Scanning electron Microscope



Figure 2 Image showing the exterior of the components of the table top SEM The components shown incorporate the concepts developed in Tasks 1.2.1, 1.2.2 & 1.2.3.

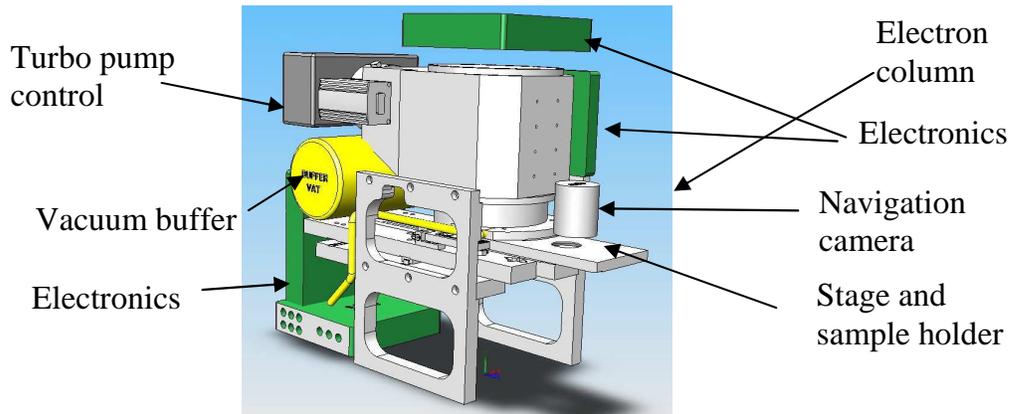
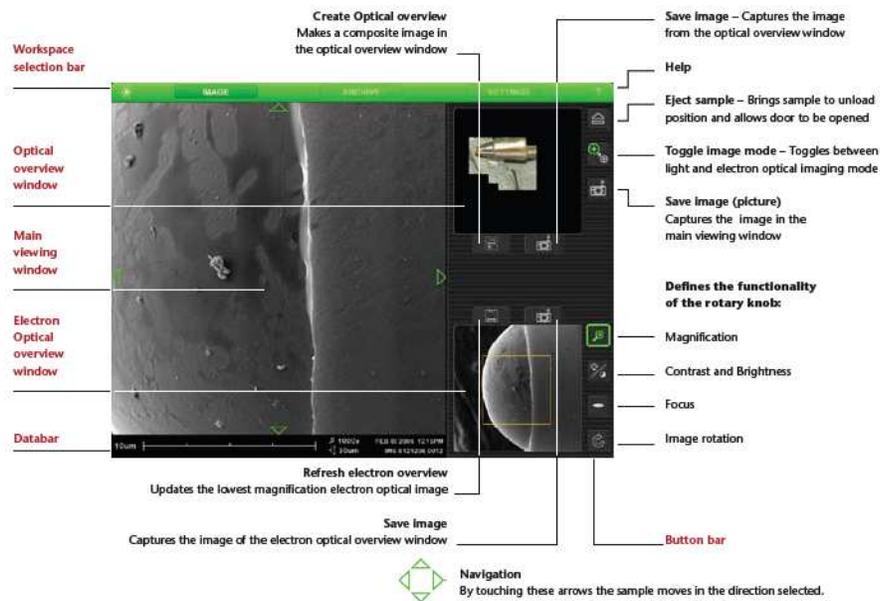


Figure 3 Image showing the main screen of the graphical user interface (GUI). The GUI shown incorporates the concepts developed in Task 1.2.4.



Task 1.4 SEM simulator

A software simulator was developed to be used in education to augment the actual hardware. The simulator was delivered to the schools and evaluated. Some of the schools found it useful, and others did not.

Task 2 Build and deliver 6 tools to schools

The tools were built and delivered to the schools for evaluation. The task was late by about 6 months, but the end date of the funded program was adjusted to allow for a complete evaluation period, as well as to ensure the full committed period of support.

Tasks 2.2-2.2.3 covered support of the tools for 12 months after tool delivery, and included support of the tools in the field by field service engineers, availability of spare parts which ensured availability and uptime, and training for both the professors and site technicians, including site manuals. This task was completed.

Task 3, Program Management

This task covered program reviews, discussions, users' group meetings, and site visits by both ONAMI and the SEM subcontractor. This task was completed without issue.

4. Identify products and technology transfer activities developed under award

a. Publications

Comparison of Materials Characterization Performed by Low Voltage Desktop SEM and Standard High Resolution SEM"
J.Lawrence, J.Carruthers, J.Jiao, and S.Berger,
Microsc.Microanal.13(Suppl. 2), 2007 1728 CD

b. Website reflecting results of this project - none

c. Networks or collaborations fostered

This project consisted of a network of universities, the funding agency, ONAMI, and a company who subcontracted to ONAMI. ONAMI organized and sponsored several regular users' group meetings, and the users shared results which were put on a sharepoint site for common use.

d. Technologies/techniques

The techniques that were transferred included applications, sample preparation, and sample handling techniques which were developed by members of the network.

e. Inventions, licensing – no patent applications resulted from this work

f. Other products including data bases, audio or video, software, educational aid or curricula, instruments, equipment

The desktop SEM simulator was completed and evaluated.
Educational aids were created using the microscope and simulator
under a follow on program funded by the BER (Appendix A).

5. **Appendix A** – BER final report of the tool evaluation (follows)

Final Report**“Tools for Nanotechnology Education Development”****Department of Energy Grant No. DE-FG02-06ER64248****Recipient: ONAMI (Oregon Nanoscience and Microtechnologies
Institute)****Principal Investigator: Robert “Skip” Rung****Submitted January 16, 2008**

I. Executive Summary

The biological and environmental research (BER) program has established long term goals which, to be successful, require detailed scientific understanding at the molecular level. State of the art electron microscopes are a key enabler in obtaining information at the nano scale and are an established technique in the BER research fields of life sciences, environmental remediation, medical applications and measurement science.

To achieve the milestones articulated in the BER strategic timeline, the United States must develop a future workforce well-trained in electron microscopy, not only for research, but more broadly, within manufacturing. Education in the use and application of electron microscopes is critical in this endeavor. Broad access to these tools is lacking in the education environment. The goal of this program was to evaluate the use of affordable electron microscopes for undergraduate education, student research experiences, and workforce training. The research on this topic will enable continued development and deployment of educational modules for continued workforce training for future nanotechnology needs.

The seven university participants in this study represented a wide diversity of student population, and the participants used a multitude of learning environments in which to test the teaching efficacy and capabilities of the affordable electron microscope. The project PI, Oregon Nanoscience and Microtechnologies Institute, Inc. (ONAMI), provided project oversight and management support to the university participants and helped facilitate exchange of information between the universities and the instrument developer, FEI Company.

II. Comparison of Project Accomplishments with Goals and Objectives

The project objectives were to develop teaching modules that require the use of a tabletop scanning electron microscope (TT-SEM) and a TT-SEM simulator that have the following key attributes:

- Easy to use by non-experts and students
- Requires no special facilities
- Affordable by postsecondary educational institutions

The expected outcome of this program is the creation of teaching modules designed to increase the pool of science and engineering majors, and better align their skills with the country's strategic needs. Prior to the start of the project, a TT-SEM and simulator were delivered to each of the seven university participants, specifically University of Oregon, Oregon State University, Portland State University, Chemeketa Community College, Winona State University, Ohio State University and Jackson State University. The instruments were developed and supported by FEI, Company, and will be marketed under the name PhenomTM.

The four overall tasks that each of the University Principal Investigators (PIs) engaged in and reported on, were:

1. Evaluation of the teaching effectiveness of the TT-SEM and simulator
2. Evaluation of the applications and capabilities of the TT-SEM and simulator
3. User friendliness of the TT-SEM and simulator
4. Quality and robustness of TT-SEM and simulator

III. Summary of Project Activities

In this section we will summarize the overall findings from the seven university PIs against the four tasks, and highlight the discussion with specific examples that help illustrate the results and activities. The actual reports are held on file with ONAMI, the principal investigator.

The original hypothesis that this program addresses is that in order to develop a workforce well-trained in electron microscopy for research and manufacturing, broad access to affordable electron microscopes must be available in the postsecondary education environment. The program was designed to evaluate the use of these electron microscopes for education, student research, and workforce training by placing tools at a wide spectrum of educational institutions, and evaluating them in a variety of learning environments.

Task 1 Evaluation of the Teaching Effectiveness of the TT-SEM and Simulator

The teaching effectiveness of the TT-SEM was universally regarded as being very high. The tool was used in a variety of settings, it engaged and excited students and teachers, and expanded the existing knowledge base of the micro and nanoscopic world. This was primarily due to the inherent resolution improvements compared to existing optical microscopy in the classroom. In particular, several professors reported the excitement that students showed when they were able to collect and image their own samples including candy surfaces, hair, and bacteria.

Portland State University (PSU) conducted a grade 9-12 “World of Nanofabrication Class” using the TT-SEM. They also used the TT-SEM in both graduate and undergraduate courses in Electron microscopy. University of Oregon (UO) took the TT-SEM to a variety of high school outreach events to “wow students and increase their interest in science and college” and reported that at one event, a low achieving female student spent a large amount of time engaged in using the TT-SEM. Chemeketa Community College (CCC), Oregon State University (OSU) and the UO kept their TT-SEM on a movable cart (Figure 1) which allowed them to use it traditional class settings, workshops, and tech fairs.

Table 1, submitted by OSU shows an example of results from a teaching effectiveness evaluation. The professor who conducted the survey said that the lower division

undergraduate students appreciated the opportunity to use a cutting edge instrument in their classes as an experiential learning event which reinforced their decisions to enter the technology based professions. The higher division students faced the use of the instrument more in the spirit it was intended; specifically as a learning tool. From the instructor's point of view, inclusion of the PHENOM in both classes is important because having an actual device which the students can use in the course of their learning is extremely important as a tool to educate visual and experiential learners, of which there are many in the engineering fields.



*Figure 1 TT-SEM on a cart for ease of portability
Courtesy of University of Oregon*

<i>Table 1. Summary of teaching effectiveness evaluations by students.</i>		
Survey Question	Course Results*	
	ENGR221	ChE417
Using an SEM was valuable part of the course	Average: 4.7	Average: 4.5
Using the PHENOM helped me to understand the difference between an optical microscope and a Scanning Electron Microscope	Average: 4.8	Average: 4.2
*Evaluated on a scale of 1 to 5, where 5 was the highest (best) possible .		

Most instructors agreed that the TT-SEM Simulator was best used for teaching large groups of students. Most students preferred a more hands-on approach using their own samples in the TT-SEM, which requires a small group setting. The teaching effectiveness

was blunted at times when the equipment failed to work properly, but this issue is discussed as a quality and robustness issue.

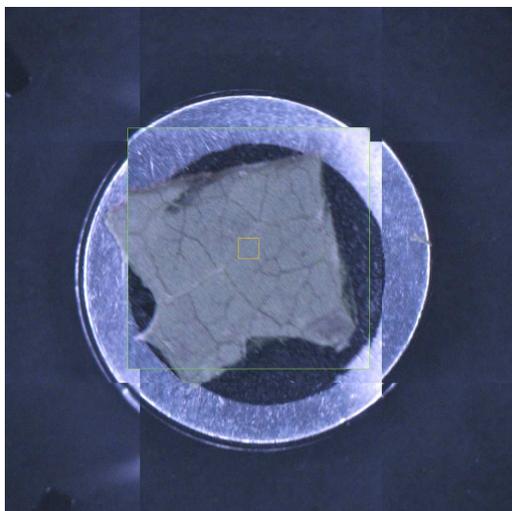
A poll taken by Jackson State University (JSU) reported that 100% of the students found the system easy to operate, increased their interest in science. 60% of JSU students polled reported that they now have a serious interest in nanoscience. Ohio State reported that they were able to use the Phenom in a number of high school outreach activities due to the ease of system use, and the high school students were capable of instructing other students as well as the Governor of the State of Ohio! Finally, Winona State University (WSU) said that use of the TT-SEM enhanced student learning by providing more structural detail (surface topographical information at the ultra-structural level) about the test organisms in the microbiology classes where it was used.

Task 2 Evaluation of Applications and Capabilities of TT-SEM and Simulator

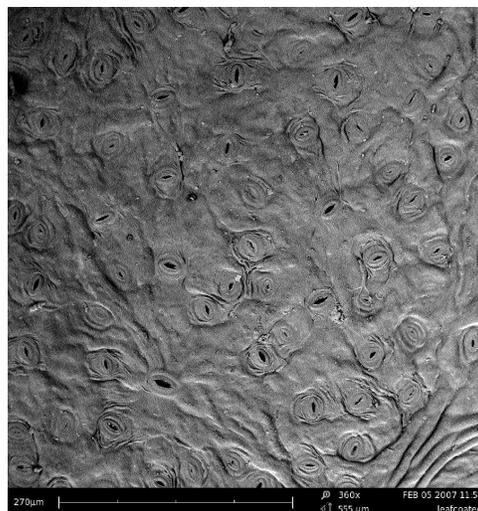
The applications and capabilities that the participating universities reported were above and beyond what was originally expected. All participants used easy to collect samples as part of applied teaching modules, as opposed to use of the TT-SEM in basic science classes. JSU developed an educational module whose learning objective was to understand the concept of an electron microscope by taking SEM micrographs. UO created high school level learning modules such as “What is on the underside of a leaf”, (Figures 2-3) and both UO and CCC created Crime Scene Investigation (CSI) style curricula. The modules can be made available by contacting the universities directly or through ONAMI.

Graduate students at the UO measured polystyrene microspheres. The TT-SEM was a part of two undergraduate classes at OSU, specifically ENGR221, “The Science, Engineering and Social Impact of Nanotechnology” and a senior chemical engineering course. The Simulator was used as part of the nanotechnology engineering class at OSU. WSU developed a histology application as part of a medical technologies curriculum: students created tissue samples from an iguana for observation with the TT-SEM. PSU’s applications were heavily biased towards nanoelectronics, including studies of silicon nanowires (Figure 4), electrodes, and palladium tubes.

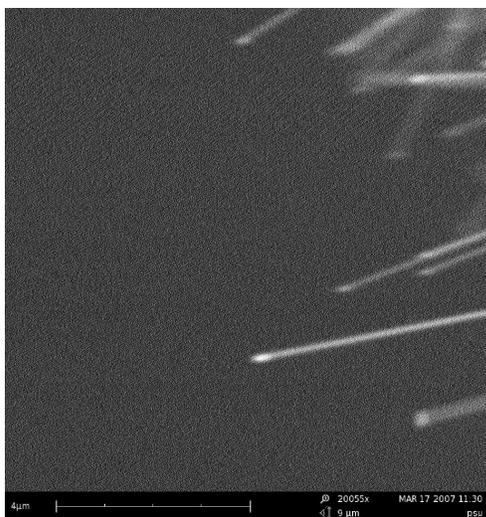
PSU also completed and published a study [*“Comparison of Materials Characterization Performed by Low Voltage Desktop SEM and Standard High Resolution SEM”* Lawrence, Carruthers, and Jiao, *Microscopy Microanalytics*, 13 (Supplement 2) 2007] comparing the resolution of the Phenom to two higher end SEMs. The general conclusions were that the Phenom has better imaging quality in the case of severely charging samples since it uses low voltage and higher chamber pressure environment. The results also suggested that the Phenom generates less obvious carbon contamination deposition during scanning due to the higher pressure in the Phenom sample chamber. This is an advantage over other SEMs.



*Figure 2 Optical image of underside of leaf
Courtesy of University of Oregon*



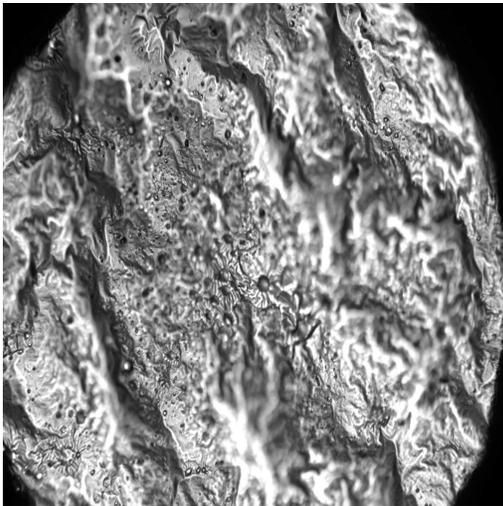
*Figure 3 TT-SEM image of leaf
555 µm Field of View
Courtesy of University of Oregon*



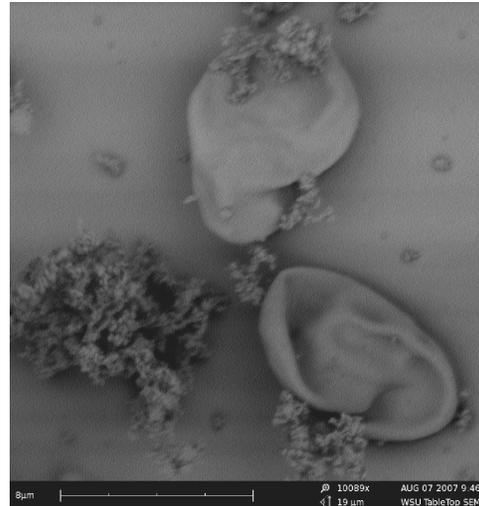
*Figure 4 Catalyst seed on end of Silicon
nanowire, 9 µm Field of View
Courtesy of Portland State University*

Task 3 User Friendliness of TT-SEM and Simulator

The user friendliness of the TT-SEM can be demonstrated by the following images taken by students at WSU in a Cell Biology class. Previously, students had only been exposed to observing animal tissue with an optical microscope (Figure 5). Using the TT-SEM, the students were able to observe microscopic cell details including red blood cells from a chicken (Figure 6) that they had not been able to observe using optical microscopy.



*Figure 5 Optical image of chicken liver
Courtesy of WSU*



*Figure 6 TT-SEM image of red
Cell from chicken liver, 19 um FOV
Courtesy of WSU*

All professors reported that the TT-SEM was remarkably easy to use and provided an enjoyable learning experience. From the UO: “Overall, the Phenom is an intuitive instrument to use. One can spend twenty minutes and demonstrate two to three samples and by then the teacher is comfortable using the machine on their own. Students seem to pick it up almost as fast, once they get over the fear of doing something wrong that will break it.” PSU reported that the majority of electron microscope users who came to use expensive, high resolution SEMs in their service lab preferred the TT-SEM because of its ease of use, and speed of image acquisition.

Some of the constructive critiques involved problems with the x-y stage mechanics and the software. Both of these problems have been corrected by the manufacturer in the released product.

WSU was the only participant that had detailed reports on the TT-SEM Simulator: “*The Simulator I also found extremely easy to use. The Phenom-Ed simulator: A guide for creating virtual samples (.smp) was very straightforward and allowed me to quickly create sample files for use with the simulator. The only part I found difficult was creation of the metadata files with the XML editor. I had never used this before. The technical support staff available at my university was able to help me pretty easily*

though. After I had completed the activity I used the Phenom-Ed Simulator User's Guide to walk me through actually looking at the images in much the same way my students would. Here I encountered no difficulties whatsoever; the directions were very easy to follow."

The other participants reported that students preferred creating their own samples in a small group setting, so each could use the actual TT-SEM. However several professors indicated that the Simulator would be most effective in a large group teaching environment.

Task 4 Quality and Robustness of TT-SEM and Simulator

All participants were provided with a beta TT-SEM which is an unreleased version of a final product. Part of the BER program was to identify the issues associated with quality and robustness so they could be addressed by the manufacturer prior to release of the final product. Thus the users were expecting 'glitches' with the equipment and reported the problems in log format to the manufacturer. These problems were repaired both in the field and at the manufacturer's site. However, the users did not like having the tool sent out to be repaired. This logistics issue was solved by offering users a loaner tool if an instrument was 'down' for an extended period of time.

The majority of the user issues involved erratic repetitive motion of the x-y stage at higher magnifications, software 'freezes' and particle generation in the vacuum chamber which caused rapid deterioration of the image. The details of the reports are maintained on file with both the universities and the tool manufacturer. The problems encountered by the users were addressed and corrected by the TT-SEM manufacturer in the released version of the SEM. The instruments belonging to each of the participating Institutions have been upgraded to include these corrections.

IV. Products Developed

The only product that was enabled by this study was the production version of the TT-SEM by the manufacturer, the Phenom, which was anticipated at the beginning of the program. (www.fei.com/Phenom.)

Various curricula modules were reported by the professors in their final reports but none were published at this time. Requests for these modules can be made directly to the universities or to ONAMI.

An informal collaboration existed between the seven universities, ONAMI and the TT-SEM manufacturer but was not formalized with legal agreements.

Publications that resulted from this study include:

"Comparison of Materials Characterization Performed by Low Voltage Desktop SEM and Standard High Resolution SEM", Juno Lawrence, John Carruthers and Jun Jiao,

Portland State University. Published in Proceedings for Microscopy and Microanalysis 2007, Microscopy and Microanalysis 13, (Suppl. 2) 2007, 1728-1729 CD.

V. Ongoing Activities

In this section we summarize activities at a few of the universities that were ongoing at the completion of this grant in October 2006.

Chemeketa Community College has created two modules in biology, titled:

- Investigation of Eukaryotic and Prokaryotic Cells using Scanning Electron Microscopy (SEM) Versus Light Microscopy (LM).
- Investigation of the Structure and Function Relationship as Related to the Ability of Certain Pollen Grains to Induce Different Allergic Reactions.

The purposes of the unit on cells are to compare and contrast eukaryotic and prokaryotic cell types, and to compare and contrast scanning electron microscopy with light microscopy. The purposes of the unit on pollen are to compare and contrast lily pollen, ragweed pollen and corn pollen with regard to pollen structure and allergy induction function, and to explain why some types of pollen are better at inducing allergic reactions than others.

Jackson State University has continued to use the Phenom in two courses

- PHY 205 “Intro to Nanoscale Science and Engineering”
- PHY 330 “Experimental Methods of Physics”

Additionally, the Phenom is an outreach tool to create excitement in pre-college students during high school student visits to JSU Dept. of Physics. Phenom images were displayed as part of an invited talk in South Africa at the International Conference on Materials Research - Aug 2007.

At **Oregon State University** the Phenom is used formally in-class in two courses

- ENGR221 (Intro to Nanotechnology)
- ChE417 (Analytical Instrumentation)

It has also been used for a class in Fracture of Materials (ME484) as well as being used as a primary sample characterization tool by a chemical engineering senior project team. Miscellaneous research groups are using the tool various chemical research disciplines, and in Outreach activities such as the “Saturday Academy” in Corvallis, Oregon. Outreach activities at “the Saturday Academy” in Corvallis (Danielle Amatore)

Portland State University reported heavy use of the tool in many areas. The Phenom was the cornerstone of a Saturday Academy short course, successfully used to train high school students. *“I liked using the microscope. It was easy to use. After someone showed me how to do it, I could run it by myself easily.”—Trina Berg, high school student*
The Phenom was used almost daily during the course of the Research Experience for Undergraduates (REU) summer program which is an NSF-funded program designed to

introduce students to research by placing them in labs for 8 weeks during the summer. Three students used the Phenom as their primary research tool

There was considerable interest from both academia and industry at the Micro Nano Breakthrough Conference, hosted by ONAMI and PSU. And, PSU conducts weekly tours of CEMN, the Center for Electron Microscopy for researchers from industry and the government.