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A Gatan Image Filter System for the Philips CM-200 FEG-TEM was installed in the Imaging and Analysis Center at Princeton Materials Institute in 1995. This addition has strongly enhanced our ability to study advanced materials by obtaining two-dimensional elemental mapping at very high resolution (nanometer range) with short acquisition times (seconds). In the past 5 years, we have made exciting progress in both research and education at Princeton and have made an impact in the materials science community by utilizing this state-of-the-art electron microscope equipped with the imaging filter system.

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FINAL PROGRESS REPORT

for the

ACQUISITION OF THE GATAN IMAGE FILTER SYSTEM AND AN ENVIRONMENTAL CELL FOR AN EXISTING PHILIPS CM-200 FEG-TEM

F49620-96-1-0405

February 28, 2001

to

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PROJECT SUMMARY

A Gatan Image Filter System for the Philips CM-200 FEG-TEM was installed in the Imaging and Analysis Center at Princeton Materials Institute in 1995. This addition has strongly enhanced our ability to study advanced materials by obtaining two-dimensional elemental mapping at very high resolution (nanometer range) with short acquisition times (seconds). In the past 5 years, we have made exciting progress in both research and education at Princeton and have made an impact in the materials science community by utilizing this state-of-the-art electron microscope equipped with the imaging filter system.

1. Research Activities

1.1 Background

Observation and discovery of new phenomena, properties, and structures are at the very heart of modern materials science and engineering. Electron microscopy, as the single, most powerful imaging and analysis technique for studying the full range of complex materials, plays an indispensable role in the field of materials science and will continue to do so well into the new century. It starts at the overview level of the optical microscope and proceeds down to the Ångstrom level of atomic structures. The electron microscope, as the second milestone after the optical microscope in microscopy history, has made great contributions to the development of science and technology in this century. Its inventor Dr. E. Ruska was awarded the 1986 Nobel Prize in Physics. Dr. Ruska developed electron optics, electron-specimen interaction theory, specimen preparation methods, and utilized electronics, precision mechanical engineering, materials science, high voltage and high vacuum technology to invent the electron microscope. A new branch of microscopy–electron microscopy–was grounded. The continuous progress over the last six decades has turned electron microscopy into an indispensable technique in materials science. Princeton Materials Institute recognized the importance of electron microscopy from its very beginning and made an immense effort to raise ~$5 million for establishing and developing a state-of-the-art electron microscopy facility at Princeton. The Durip's award for the acquisition of the Gatan Image Filter System has been a big step forward to improve the imaging and analysis capability at atomic scale at Princeton.

Our research has been focused on using advanced imaging and spectroscopy techniques to conduct fundamental studies of the structure-composition-processing-property relationships in complex materials such as self-assembled organic/inorganic composites, functional block copolymer thin films, carbon nanotubes, superconductors, minerals, ferroelectric nano-crystallites, and other materials. Since 1996, we have authored ~75 research papers and 3 patent disclosures, including 3 articles published in *Nature* or *Science*, which have been

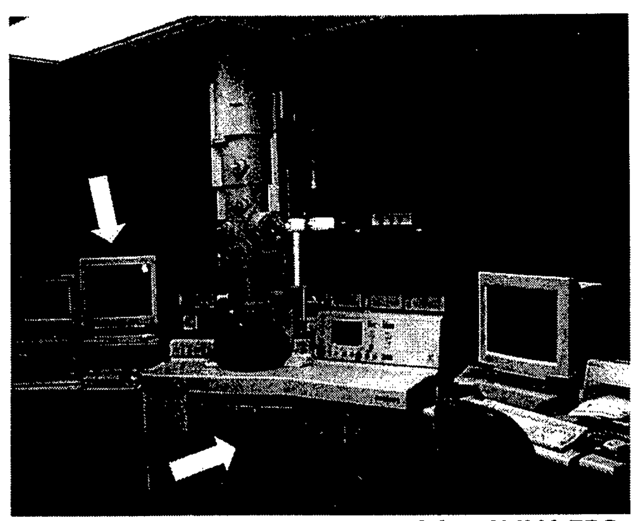

***Figure 1**. The Princeton University's Philips CM200 FEG-TEM equipped with the Gatan Image Filter (GIF200) system (see arrows)*

cited over 270 times by peers in the past three years.

1.2 Multidisciplinary Research within Princeton

Many research groups both within and outside of Princeton University have utilized the Philips CM200 FEG-TEM equipped with the imaging filter system. Users at Princeton include: I. A. Aksay, W. B. Russel, R. A. Register (PMI and Chem. E.), A.G. Evans, (PMI and MAE), S. R. Forrest (PMI and E. E.), J. C. Sturm, S. Y. Chou (E. E. and POEM), P. M. Chaikin, N. P. Ong (Phys and PMI), R. J. Cava (PMI and Chem.), G. W. Scherer (PMI and CE), G. C. Dismukes, M. H. Hecht, J. T. Groves (Chem), E. Wieschaus (Mol. Bio.), and D. R. Mumm, N. Yao (PMI). The new capabilities provided by this instrument have also strengthened our on-going collaboration with local industry (Exxon, NEC, Johnson & Johnson, Merck, and Engelhard). The major research projects include:

(a) Research in Carbon Nanotubes

As a new class of nanostructured materials, carbon nanotubes exhibit physical and chemical properties that are remarkably different from other materials. These tiny nanotubes are expected to have great potential for applications including "one-dimensional" wires, single-molecule transistors, minuscule field-emission electron guns, and tiny devices. Collaborating with undergraduate student Vincenzo Lordi* (ChemE '99) and NEC scientists, we have made exciting progress in this research area. Related results are detailed in 12 research papers recently, we have developed a new carbon nanotube related research project for a senior thesis. The research topics include:

- Discovered novel mechanical properties of carbon nanotubes. Showed that unlike other materials, carbon nanotubes are so small that changes in structure can affect the Young's modulus. We reported for the first time the exceptional variation of Young's modulus of individual single-walled and multi-walled carbon nanotubes with tube radius and helicity.
- Studied the dynamics of nanotubes under axial compressive stresses. Demonstrated that a nanotube cap can act as the world smallest Hookean spring – a potential imaging probe for studying the atomic structure of materials surfaces.
- Investigated nanotube response to asymmetrical radial "cutting" forces and the feasibility of mechanically cutting individual nanotubes. We showed that the magnitude of distortion

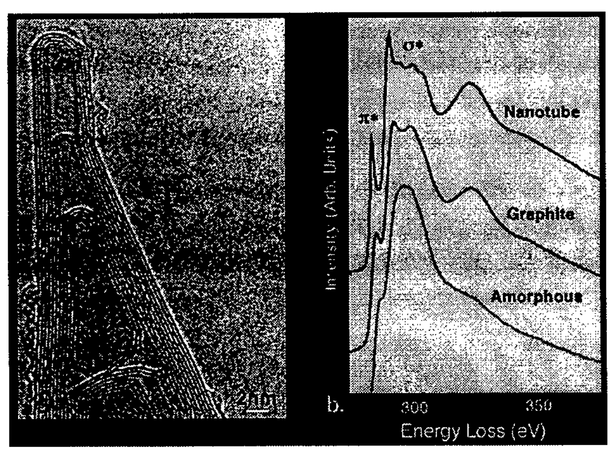

Figure 2*. (a) High resolution Transmission Electron Microscopy image of a Multi-walled carbon nanotube tips, and (b) the electron energy loss spectra showing the characteristics of carbon nanotubes, graphite and amorphous carbon.*

*Vincenzo Lordi has won one of five 1998 Undergraduate Research Awards from the Microscopy Society of America in recognition of his research achievements and excellent academic standing. He also won the prestigious Hertz Fellowship in 1999, which is awarded to only 20 students annually nationwide.

force can readily be estimated and is related to the shape of the distortion, characterized by percent compression and radius of the tube. The magnitude of the force also depends on the number of layers for very narrow tubes.

- Described in detail the relationship between pentagon positions, helicity and the percentage of zigzag sites on the carbon nanotube edge. Demonstrated that the nanotube oxidation process can be used to probe individual pentagons, strain locations, and helicity of individual shells in carbon nanotubes.
- Conducted a systematic study on the role of support on catalytic activity and selectivity. Detailed investigation of Pt particle bonding to oxidized single-walled carbon nanotubes.

(b) Research contributions to NSF MRSEC program

In addition to the carbon nanotube research described above, we have worked with researchers from a wide range of disciplines at Princeton. We have interacted with 44 faculty members, over 120 students and post-docs from 10 departments (Chemical Engineering, Chemistry, Geological and Geophysical Sciences, Physics, Molecular Biology, Electrical Engineering, Mechanical and Aerospace Engineering, Princeton Plasma Physics Laboratory, Ecology and Evolutionary Biology, and Civil Engineering and Operations Research).

Particularly, we have devoted our major research effort in areas initiated by other faculty members and have played a leading role in a number of research projects. We have been leading authors for ~20 papers among 50+ collaboration related publications. We made contributions to all four Integrated Research Groups (IRGs) under the Materials Research and Engineering Center (MRSEC) program funded by the NSF. Detailed list of research area follows below:

IRG 4-Design and Processing of Bioinspired Composites

Extensive research collaboration with research groups led by I. A. Aksay, J. T. Groves, S. M. Gruner. This collaboration has produced more than 30 publications (including 3 articles published in *Science* and *Nature*. Our research results have also been used to support a number of proposals for future research funds. Major projects include:

- Developed a new method to form mesoscopic silica thin films via template-assisted self-assembly. Brought together the evidence from the recent materials science literature linking the crystal morphogenesis, nano-scale structure, and acidic conditions. Provided new insights into the interface interaction and a new nucleation and growth mechanism for synthesizing mesoscopic silica thin films through supramolecular-template-assisted self-assembly.

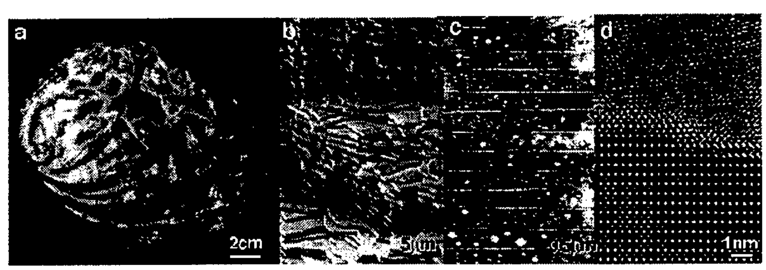

Figure 3. *A series of images showing the structure of an Ablone shell. (a) Outlook of a shell structure; (b) An SEM image of fracture surface of the shell interior structure; (c) A TEM image revealing a <10nm thin organic film between the aragonite platelets; and (d) a High-resolution atomic TEM image showing an interface between organic layer and aragonite plate.*

- Developed a method that imposes a hierarchy of microscopic structures on thin solid films by combining a surfactant self-assembly for templating nanoscale channels in silica with a micromolding technique. Although HREM techniques have developed significantly in recent years, high resolution imaging of soft materials remains a tedious and difficult task. We have utilized the ultramicrotome technique to improve ultrathin cross-sections of a surfactant-silicate film on a solid substrate for HREM analysis.

- Developed silicified L_3 phase materials with continuously adjustable pore sizes >10nm using surface reaction of a surfactant L_3. Using ultramicrotome sample preparation and low temperature (LN_2) TEM technique we have conducted the structure characterization to identify this new material.
- Demonstrated for the first time that an anionic porphyrin can assemble into a biomimetic nucleation template. Developed a method to synthesize macroscopic-scale continuous $CaCO_3$ thin films. Provided new insights into the template-inhibitor-biomineral interaction and a new mechanism for synthesizing ceramic thin film under mild conditions.
- Developed a biomimetic method using self-patterned triblock copolymer as template to form a barium titanate/polymer composite.
- Using High-resolution electron imaging and chemical analysis, identified for the first time the co-existence of two mullite phases (3:2 and 2:1 phase) in nanometer scale. This discovery is crucial for understanding the phase evolution during low temperature mullitization.

  Other projects are:

  - HREM study of the breakup of intermediate gold nanocrystallites.
  - Liquid phase sintering of BN doped Fe-Cu/TiC composite.
  - The effect of additives on the silicification of wood cell walls.
  - The role of the trans-phase interfaces on the mechanical properties on SiC-mullite-Al_2O_3 nanocomposites.
- Research collaboration with Alex Navrotsky on HREM and convergent beam electron diffraction (CBED) studies of ordered high-pressure perovskites and titanium dioxide-surfactant mesophases and Ti-TMS1. We determined the structure, symmetry and local composition variation of a new perovskite, $CaFeTi_2O_6$, from a sub-micrometer single crystal. Reported the first evidence of four types of superstructure in Ca_2TiSiO_6 perovskite.

IRG-3 Amphiphilic Polymers: Functional Materials Through Self-Assembly

Research collaboration with research group led by P. Chaikin, R. Register and W. B. Russel. Working closely with graduate and post-doc researchers: P. Mansky, C. Harrison and M. Park in the group. Research projects include:

- Developed a new imaging method for characterizing surface structure. By controlling the electron penetration depth in the low energy range in a SEM, surface sampling depth information can be obtained within the nanometer regime [Ref. 39,40]. With this method, the diblock copolymer thin film can be imaged in a layer-by-layer fashion.
- This new low-voltage imaging technique, together with other electron microscopy methods, has been effectively used in developing diblock copolymer monolayer thin film as

nanolithographic masks. This technique accesses a length scale difficult to produce by conventional lithography and opens a route for patterning of surfaces via self-assembly.

IRG-2 Organic thin films and Quantum Structures

Research collaboration with research group led by S. R. Forrest, P. E. Burrows, and M. E. Thompson. Research topics include:

- Intense second harmonic generation and long-range structural ordering in thin films of an organic salt grown by organic vapor phase deposition.
- Studies of temperature effect on Alq3 film growth.
- Synthesis and study of zirconium viologen phosphonate compounds on a polymer surfactant template with colloidal Pt particles.

IRG-1 Unusual Phases and Excitations in Low Dimensional Electron Systems

Research collaboration with group led by N. P. Ong, P. Anderson, and K. Molar, Projects include:

- Structure of $Tl_2Ba_2CuO_{6+\delta}$ (2201) crystals.
- Studies of Zn and Gd doping in the $YBa_2Cu_3O_{7-x}$ (123) superconductor.
- Studies of Y doping in the $Bi_2Sr_2(Ca_{1-x}Y_x)Cu_2O_8$ (2212) superconductor.

(c) Research interaction with other research groups

- We have also interacted with 33 other groups on electron microscopy related projects, including groups led by, respectively, A. Bocarsly (Chem.), J. C. Carey (Chem.), C. Dismukes (Chem.), F. Bracco (MAE), J. B. Benziger (Chem. E.), W. Happer (Phys.), P. Heaney (Geo.), M. Hecht (Chem.), H. Horn (EEB), G. Keller (Geo.), F. Morel (Geo.), G. Scoles (Chem.), A. Kahn (E. E.), P. Jeffy (MAE), T. Onstott (GEO), R. K. Prud'homme (Chem. E.), S. Chou (E. E.), S. Wagner (E. E.), B. Royce (MAE), M. Goeckner (PPPL), G. Scherer (CEOR), E. F. Wieschaus (Mol. Biology), G. M. Schupbach (Mol. Biology), J. C. Sturm (E. E.), M. Shayegan (E. E.), S. Sundaresan (Chem. E), Z. G. Suo (MAE), S. Torquato (Civil E.), J. Turkevich (Chem. Emeritus), W. Warren (Chem.), L. Sohn (Phys), B. Keimer (Phys.), and R. Cava (Chem.).
- Using high-resolution electron microscopy, with J. B. Benziger and S. Sundaresan we have investigated the evolution of the active surface of the vanadyl pyrophosphate catalyst.
- The Princeton Materials Fair has become a major annual event to evaluate the on-going research activities at PMI. Our contribution has been represented by a total of 60 posters in the past five years.

1.3 Research Interaction with Industries

Industrial collaboration can not only promote scientific exchange, but also establish our EM facility as a leading characterization center in the materials research community. We have been actively interacted with 32 scientists in 18 industrial companies, as shown in Table 1.

- Developed Pt based model catalysts with a sol-gel method for the studies of effects of metal location and loading on both inner- and outer surface of oxide carrier.

Table 1. IAC Industrial Collaborations

A. Litwak, C. Wang (Therics, Inc.)	C. Hahn (Nalco/Exxon)
D. Denley, G. York, and M. Bolinger (Shell)	F. Allen, I. Petrovic, G. Munzing (Engelhard Co.)
G. Abrahan (Revlon)	I. Irush (Princeton Instruments)
J. Higgins (Mobil)	M. Lund (MOXTEK, Inc)
M. M. Disko, W. A. Lamberti, J. H. Sinfelt (Exxon)	R. Kemp (Union Carbide Co.)
T. W. Ebbesen, D. Norris, E. Dujardin, A. Krishnan, P. Fisher, M. M. J. Treacy, T. Thio, D. Grupp (NEC)	X. Tang (Rohm-Haas)
R. B. Marcus (Murry Hill Device)	K. Zhang (Princeton Electronic Systems)
Z. G. Li (duPont)	Y. Gao, J. Chun (FMC)
G. Smelik (Molten Metal Tech., Inc.)	L. Lin (AT&T Labs)

- Developed a method to use oxygen/carbon reaction as an atomic probe to study carbon nanotube structure.
- Developed a method to form 3-D patterned semiconductor using densely packed CdSe quantum dots, using a synthetic opal as a template. This provides a general route to both quantum-dot solids and polycrystalline semiconductors that are patterned in three-dimension.

1.4 Patent Disclosures Made Since 1996

- "Biometic Pathways For Assembling Inorganic Thin Films", Inventors: I. A. Aksay, M. Trau, S. Manne, I. Honma, N. Yao, L. Zhou, P. Fenter, P. M. Eisenberger, and S. M. Gruner.
- "Formation of a Silicate Sponge (L_3) Phase with Continuously Adjustable Pore Sizes," Inventors: K. M. McGrath, D. Dabbs, N. Yao, I. A. Aksay, and S. M. Gruner.
- "The world smallest springs: carbon nanotube caps", Inventors: N. Yao, V. Lordi.

2. Educational Activities

We have used this instrument to introduce imaging and analytical techniques to students from various classes including MSE300, ELE 542, GEO 312, GEO 514 and MSE505. Our short course in Transmission and Scanning Electron Microscopy has drawn over 100 students for 5 weeks of lecture and lab during each summer over the past six years. The student enrollment continually increased from 9 in 1995 to 25 in 1998. We also supervised over 20 undergraduate student theses.

The multidisciplinary nature of PMI requires me to play an active role in the materials science education. In addition to the research and administration responsibilities, We have conducted various microscopy teaching activities including: a) short course in electron microscopy, b) MSE 300, c) lecture in ELE 542, GEO 512, and GEO 312, d) co-advised 8 undergraduate thesis, e) lectured and trained a total of 300 students and researchers on using electron microscopes and related instruments.

- PMI has regularly offered a short course on the applications of electron microscopy to materials science during each summer in the past 6 years. This course has drawn a total of over 100 students (9 in 1995, 14 in 1996, 18 in1997, 20 in 1998, 20 in 1999, and 19 in 2000) for 5 week's lecture and lab. Students comprise undergraduates, graduates, post-docs and research scientists from 8 departments (CEOR, Chem., Chem. E., E. E., Mol. Bio. Phys., Geoscience, and PPPL). This course has accomplished two goals: (1) introduce students to the fundamentals of both transmission electron microscopy (TEM) and scanning electron microscopy (SEM) techniques, and (2) provide students with the requisite knowledge and skills to operate and obtain meaningful results from modern TEM and SEM. It covered general aspects of TEM and SEM emphasizing hands-on experience and interpretation of images. Some specimen preparation techniques such as ion milling and ultramicrotome were also included. Extensive hands-on operation was provided to every student.

  This short course has been well received by students. The feedback from students (see Appendix B) shows the satisfaction level for the course is high. Students who took this course have applied electron microscopy techniques in their research, and they dominated the PMI Materials Fairs Presentation Contests. Winners include: J. Lahiri (Chem., 1994), T. Lee (Chem. E., 1995) and G. F. Xu (Chem., 1995), and B. De (Geo. -Phys. 1996)

- Taught Electron Microscopy in PMI's MSE 300 course (four week's lecture and lab, a total of 66 students enrolled, 17 in '93, 8 in '94, 6 in '95, 10 in '96, 18 in '97, and 7 in '98) in the past six years. Our lecture was described as "the best lecture I have ever had" by Daniel Katz (Chem. '97, Valedictorian).
- As an invited instructor participating in teaching ELE 542, GEO 312, GEO 512 courses, lecturing on Transmission Electron Microscopy and Scanning Electron Microscopy.
- Selected by students to co-advise three undergraduate senior thesis.
- Selected by students to advise six undergraduate junior projects (JP) and other research projects.
- We have also taught more than 80 researchers through more than 40 training sessions on how to use the CM200 FEG-TEM, Zeiss 910TEM, Ultramicrotome, and FEG-SEM. They are now extensively utilizing electron microscopes in related research.
- Taught and supervised more than 50 students on using Cerius^2 molecular modeling and simulation software on SGI Indy workstations for research.
- Presented tutorial lecture on electron microscopy to P. Anderson and K. Moler of Physics.
- Guided and worked with the following 15 undergraduate students in electron microscopy related research projects.

3. The Imaging and Analysis Center development

The Princeton Materials Institute (PMI) established the Imaging and Analysis Center (IAC) in 1993 to promote multidisciplinary collaborations in research and education and to strengthen efforts in materials science and engineering by providing critically needed central facility. The

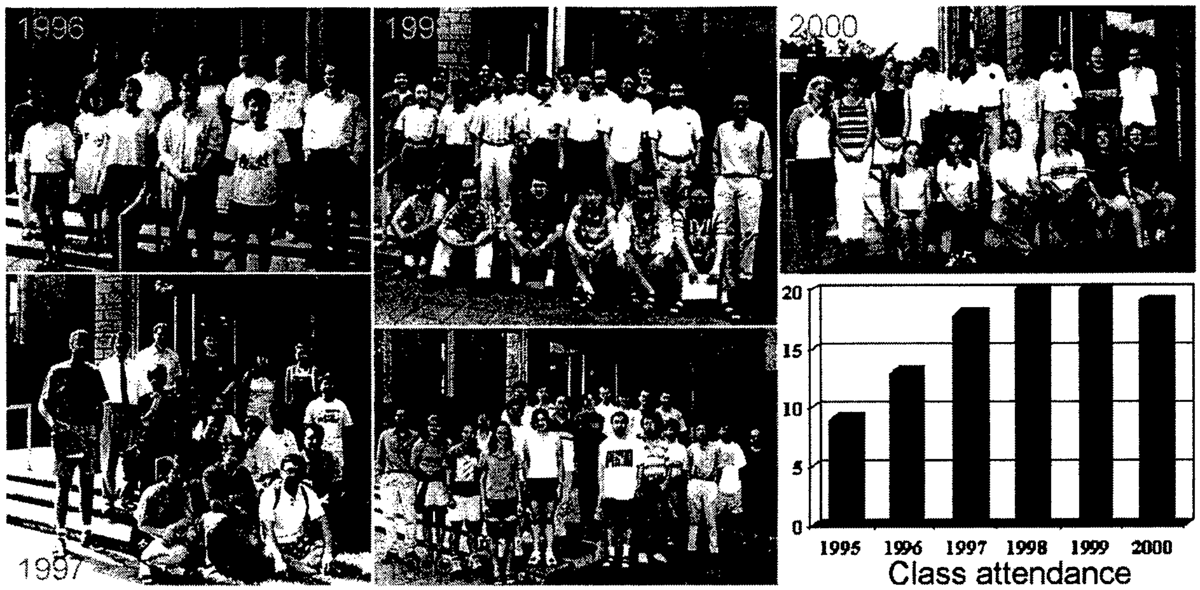

Figure 4. PMI Electron Microscopy Summer class photographs, 1996-2000.

IAC is a major imaging and analytic instrumentation center, which is supported by both PMI and the Princeton Center for Complex Materials (PCCM). Since its inception the IAC has occupied a pivotal role in materials research and education at Princeton. With continued funding support from NSF, AFOSR, ONR, the state of New Jersey, Exxon, Johnson & Johnson and the University, the IAC has become one of the most advanced imaging and analysis facilities in the world.

The IAC currently comprises advanced electron microscopy techniques, especially transmission (a Philips CM200 FEG-TEM and a LEO/Zeiss 910 TEM) and scanning (a Philips XL30 FEG-SEM and a LEO 982 FEG-SEM). This provides high-resolution, energy-filtering and low-temperature TEM, nano-diffraction EM, low-voltage SEM and in situ mechanical testing capabilities. It also has microchemical and microstructural analysis capabilities (EDS, PEELS, WDS, CL, OIM), probe microscopes (a Cameca SX 50 electron microprobe, DI AFMs, and a UHV-STM), computer simulation (molecular dynamics and image processing), and materials preparation (ion beam sputtering, cryo-ultra-microtome, mechanical dimpler, and ion beam mill). More recently, a new Dual-beam Focused Ion Beam System is being added in the Center. With its precision of ion-beam milling, high-resolution imaging, and gas chemistries, this new instrument provides the power and flexibility for full 3D structural analysis and characterization of sub-0.13 micron processes, including copper dual damascene, low-k dielectric, chemical mechanical polishing, and high-aspect ratio structure.

4. Educational Outreach Activities

The IAC supports the research and educational activities of the faculty and students at PMI and PCCM and more broadly at the University. It also collaborates with researchers in industry. The IAC's users include 50 faculty, involving 150 students and post-doctoral fellows, from 10 Departments [Chemical engineering, Chemistry, Electrical Engineering, Mechanical and Aerospace Engineering, Geosciences, Physics, Molecular Biology, Plasma Physics (PPPL), Ecology and Evolutionary Biology, and Civil Engineering], as well as about 30 industrial scientists from 18 corporations: including Exxon, NEC, Engelhard, Revlon, FMC, duPont, AT&T, Merck, etc. About 120-refereed papers related to the IAC have been published in the past two years. IAC has also been a major facility for the undergraduate and graduate students. Students from MSE 302, MSE 505, CEE 105, ChemE 346, Chem 511and GEO 512 enjoy using various types of electron microscopes during class, as well as in their independent research and senior theses. The research experience at IAC has helped them win a number of national awards, such as John Hertz Fellowship, LeRoy Apker Award, Microscopy Society of America Undergraduate Research Award, National Science Foundation Fellowship, National Defense Science and Engineering Fellowship, Materials Research Society Student Award, etc. Many REU and RET members have also learned to use the instruments during their stay at Princeton. Over the past six years, the IAC's summer electron microscopy course for graduates and Post-docs has drawn a total of over 110-student enrollment.

5. Publications

1. N. Yao, R. Z. Wang, A. Y. Ku, D. A. Saville, and I. A. Aksay, "Nanostructured Bio-inspired Materials", a chapter in the book entitled "Nanophase and Nanostructured Materials", edited by Z. L. Wang, Y. Liu and Z. Zhang (Tsinghua University Press and Wiley-VCH) (2001) in press.

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