

# LeRoy Eyring Center for Solid State Science

Nate Newman, director

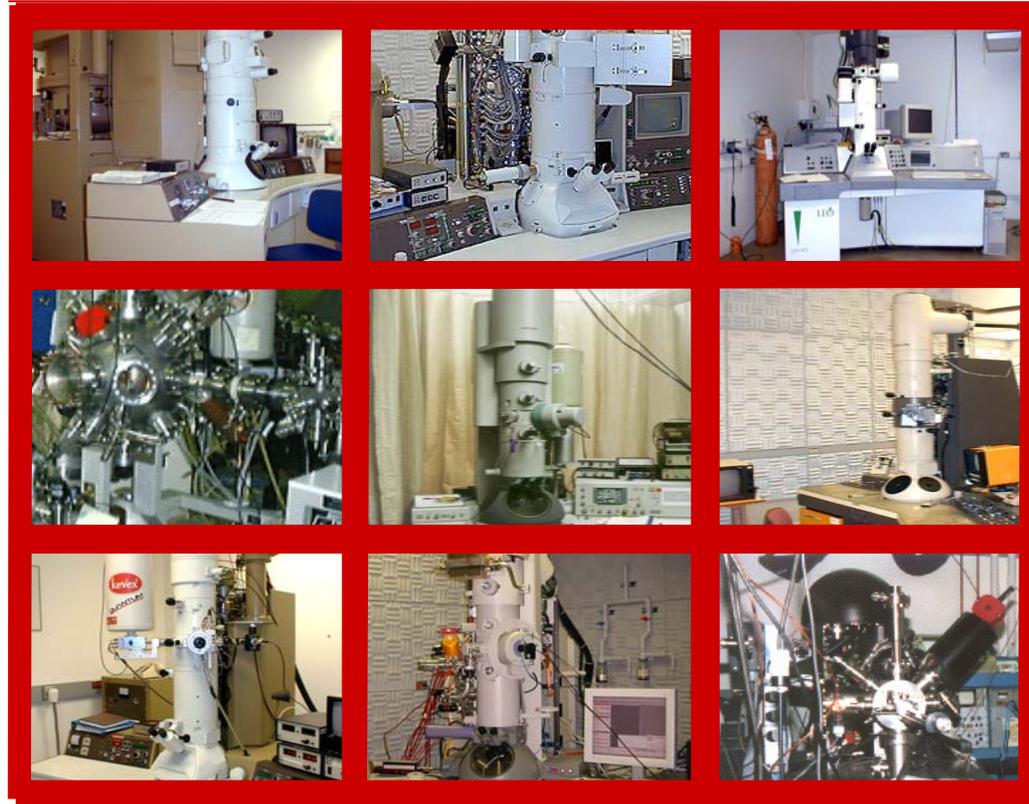
- Established by ABOR on May 25, 1974  
encourage & support interdisciplinary research activities in solid state materials
  
- Highlights of current status
  - \* >\$2+ Million facility budget - 20% higher than our last year record value
  - \* >\$32 M “touched-view” of research expenditures
  - \* >40 instruments available to ASU & outside research community including TEM, SEM, FIB, SQUID magnetometer, RBS, AFM, AES, XPS, thermochemical analysis
  - \* >200 current users, >70 P.I.s, >20 departments and >4 colleges
  - \* >Outreach- “*Science is Fun*” program reaches >20,000 K-12 students
  - \* > Members of IAP include Intel, Chevron, Monsanto, Dial, Freescale, .....
  
- 1200 users to-date, our most important “product”

# Major objectives of the Center are:

- to encourage and support interdisciplinary research activities in solid state science
- to operate & administer user-facilities for multidisciplinary research
- to support outreach activities ; and
- to provide a valuable resource for industry through industrial affiliates programs

# Expensive Advanced Instrumentation

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Milions \$\$\$\$ invested in instrumentation (unique)

Thousands of hours in associated expertise

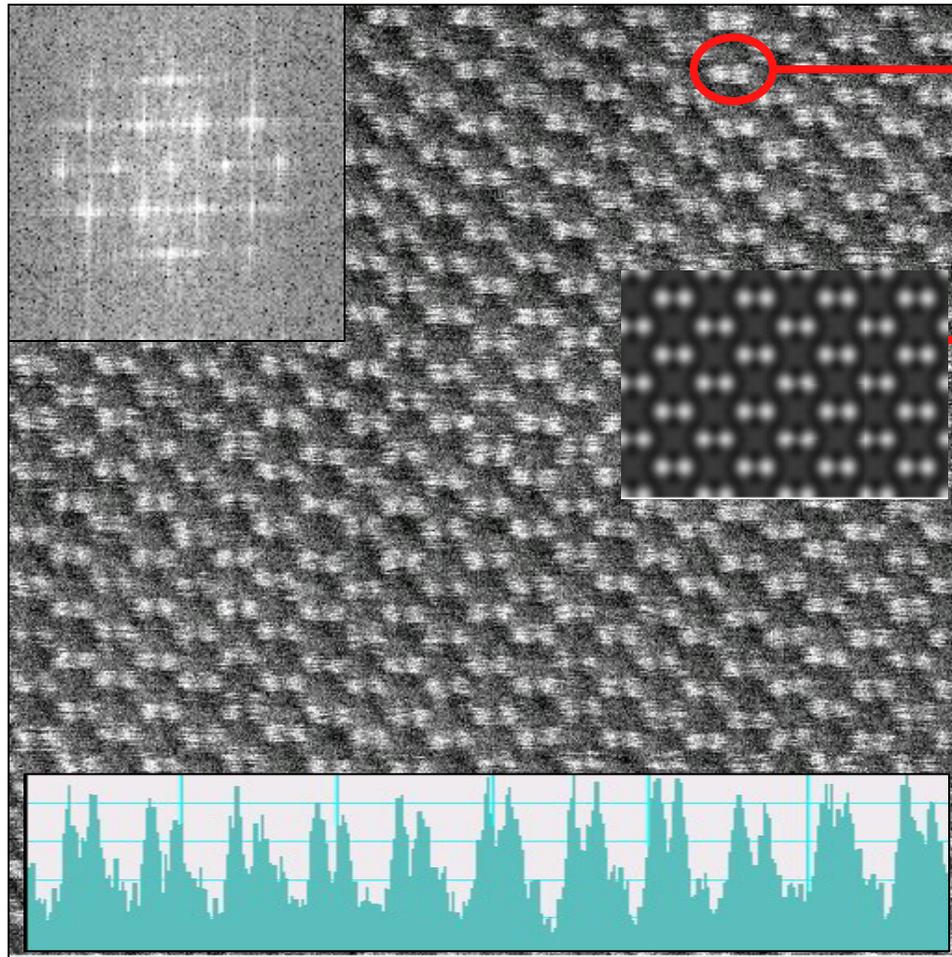
# People who make it happen

## LE-CSSS Staff

<b>1984-present</b>	<b>Karl Weiss,</b>	<b>23 years</b>
<b>1985-present</b>	<b>Renu Sharma</b>	<b>22 years</b>
<b>1985-present</b>	<b>Bonnie Mello</b>	<b>22 years</b>
<b>1985-present</b>	<b>David Wright</b>	<b>20 years</b>
<b>1987-present</b>	<b>Peter Crozier</b>	<b>20 years</b>
<b>1987-present</b>	<b>Paul Perkes</b>	<b>20 years</b>
<b>1989-present</b>	<b>Timothy Karcher</b>	<b>18 years</b>
<b>1994-present</b>	<b>Barry Wilkens</b>	<b>15 years</b>
<b>2000-present</b>	<b>Zhenquan Liu</b>	<b>7 years</b>
<b>2000-present</b>	<b>Jon Mull</b>	<b>7 years</b>
<b>2002-present</b>	<b>Grant Baumgardner</b>	<b>5 years</b>
<b>2003-present</b>	<b>Donalea Robertson</b>	<b>3/7 years</b>
<b>2003-present</b>	<b>Joanne Ackerman</b>	<b>3/14 years</b>
<b>2007-present</b>	<b>Sisouk "Si" Phrasavath</b>	<b>1 year</b>
<b>2007-present</b>	<b>Roxanna montoya</b>	<b>3 days</b>

# Atomic Resolution Z-Contrast Imaging

*Z-contrast image of Si <110> orientation – atoms are white*



*Separation of pair  
of Si atoms ~ 0.14 – 0.15nm*

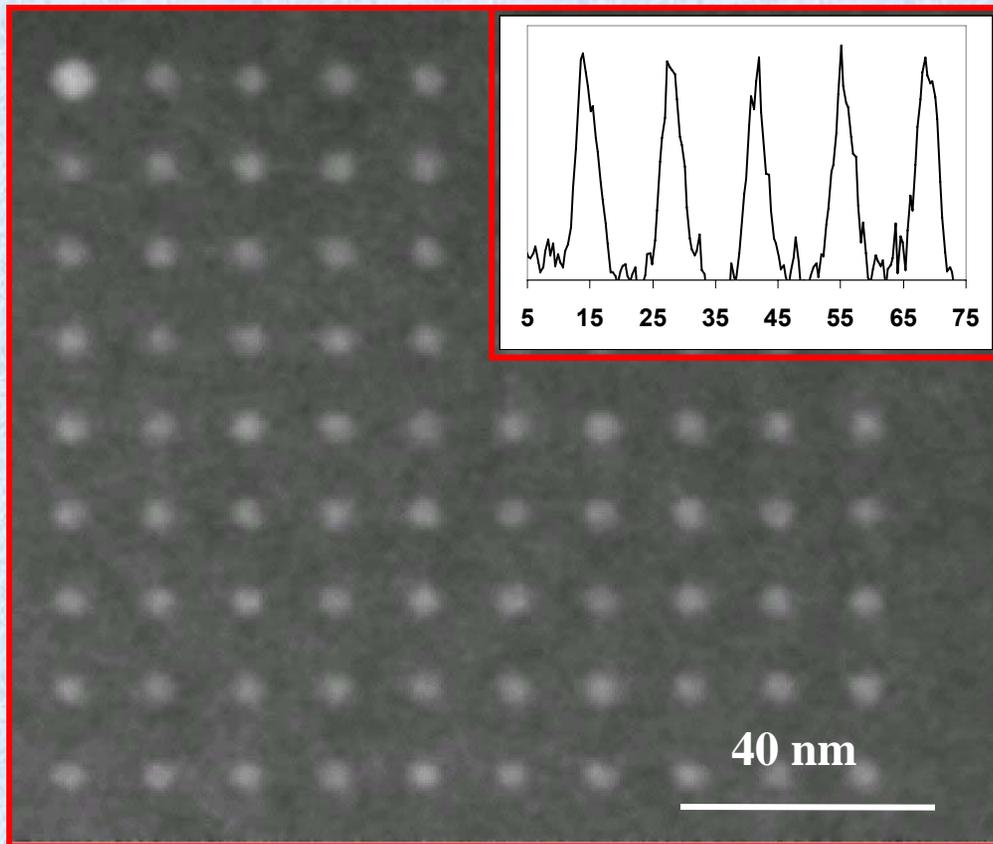
*Z-contrast image  
simulation conducted  
using Kirkland codes  
 $\Delta f = 50 \text{ nm}$ ,  $t = 5 \text{ nm}$*

E.J. Kirkland – Advanced Computing for  
Electron Microscopy 1998

*For thin samples,  
focusing is unique  
no contrast reversals !*

# *Ordered Arrays of GaN Dots on Si*

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❖ *10 x 10 array drawn from adsorbed layers*

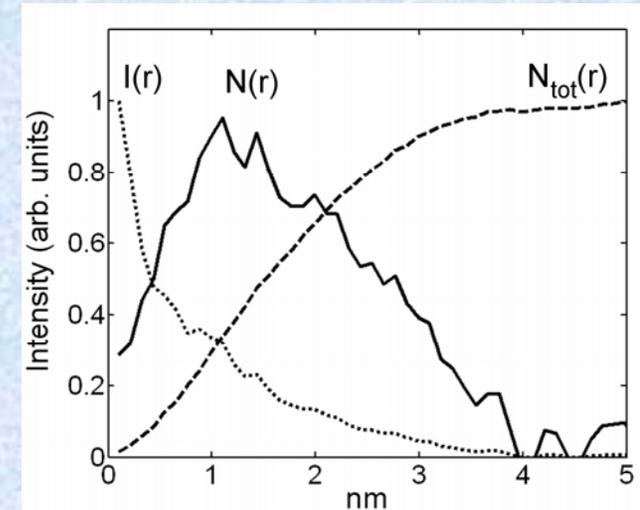
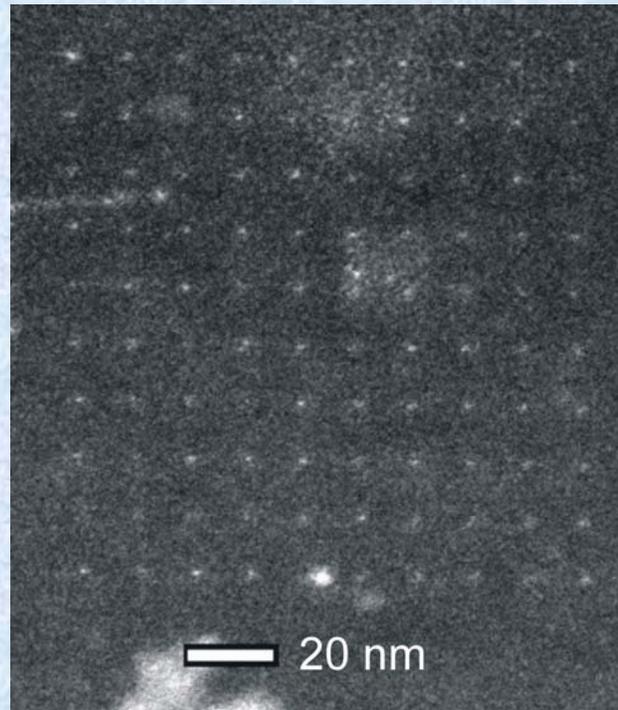
❖ *GaN dots are very regular and show uniform size with base width of 9 nm and FWHM of 4 nm.*

# Decomposition of $W(CO)_6$

Array of very  
small  $W$  dots  
on  $Si_3N_4$

Very few atoms  
in dot ( $\sim 10 - 15$ )

Shot noise leads  
to fluctuations

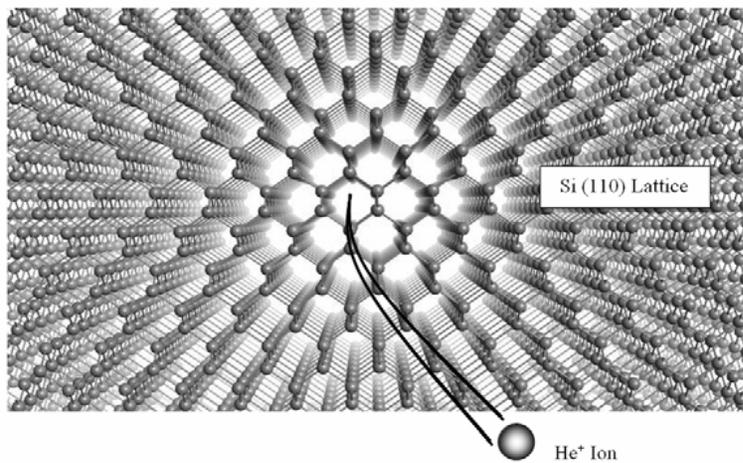


*FWHM of average 1 nm - a world record for EBID !!!!*

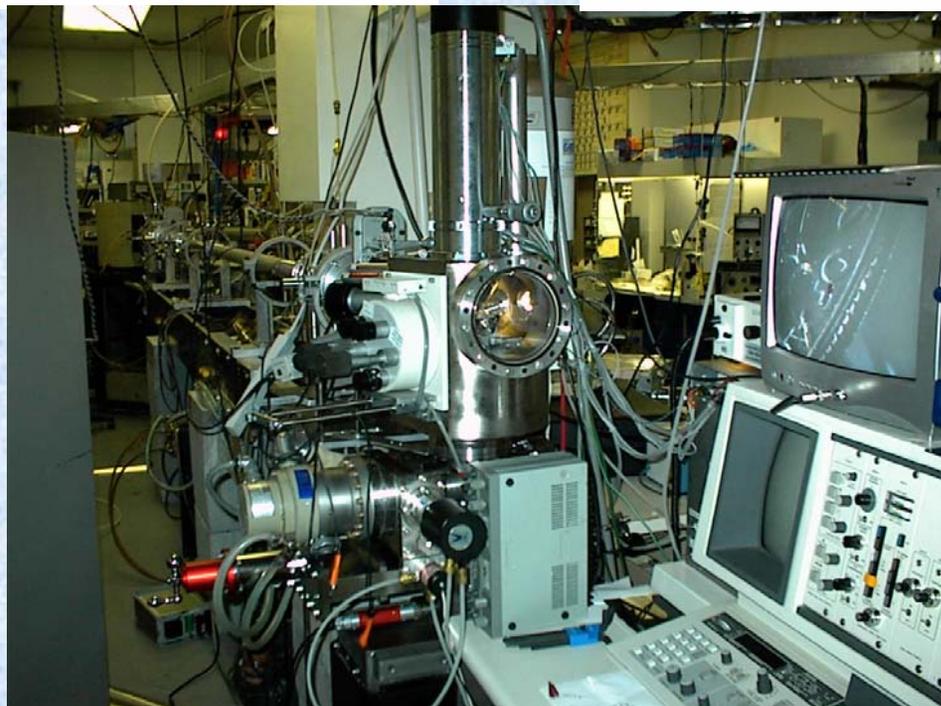
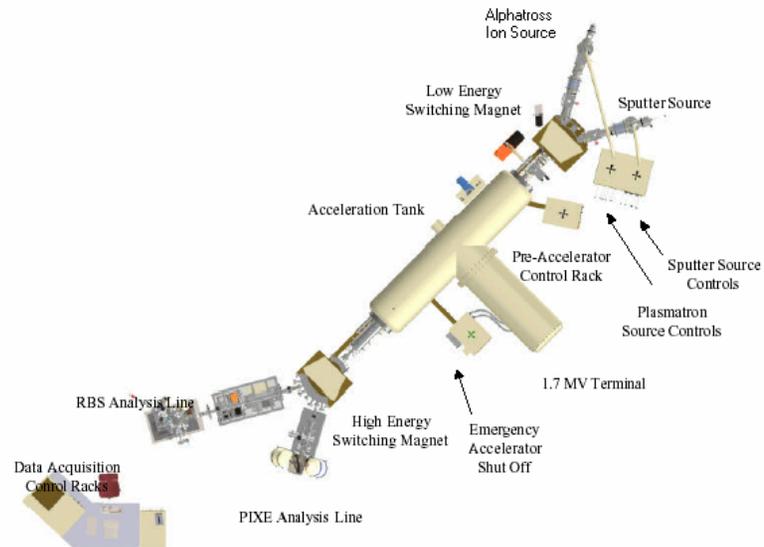
*Smallest cluster is 0.7 nm*

*(submitted to Nanoletters)*

### Ion Channeling



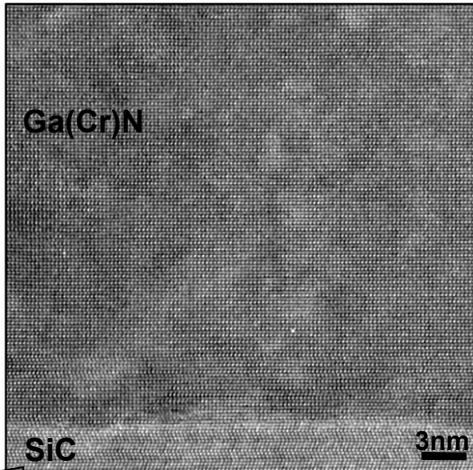
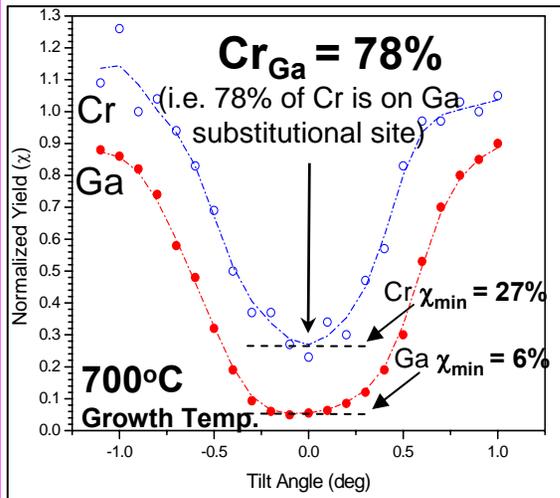
### Accelerator Overview



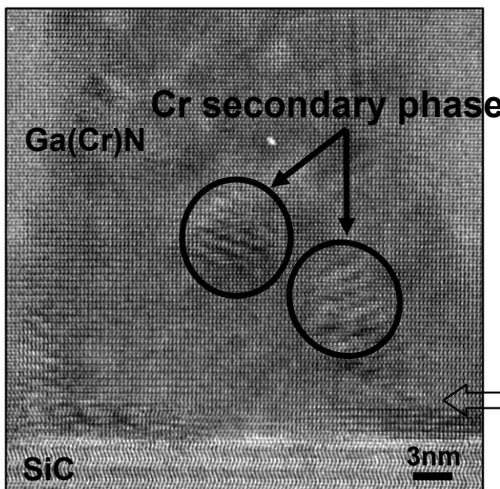
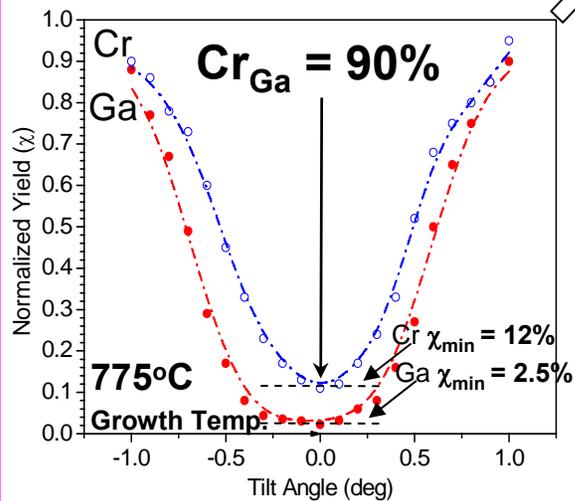
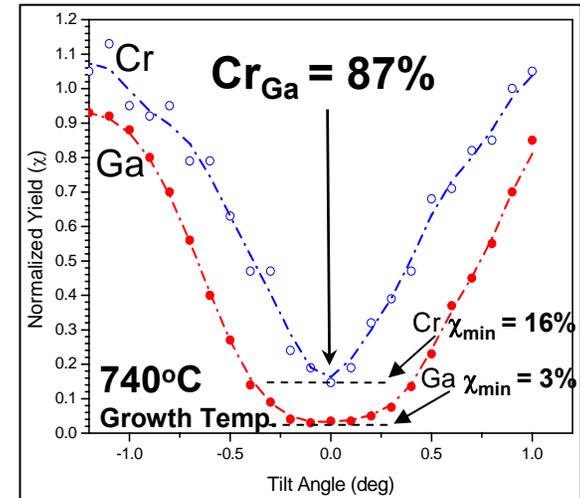
# Ion-channeling RBS:

## Measured Angular Distribution for 3% Cr-doped GaN

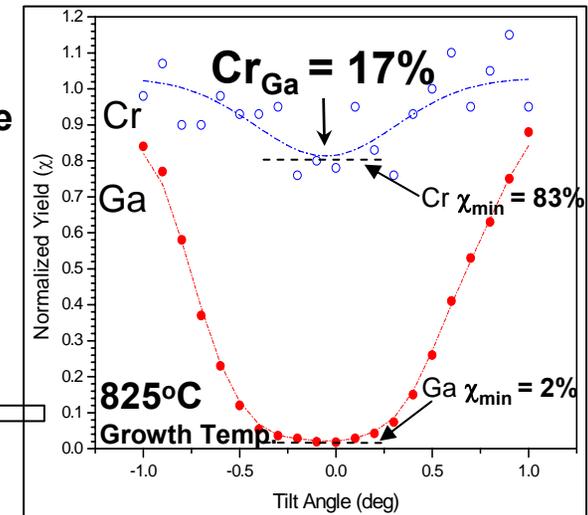
Low Ga  $\chi_{\min}$   $\rightarrow$  excellent crystallinity; Low Cr  $\chi_{\min}$   $\rightarrow$  high  $\text{Cr}_{\text{Ga}}$  (Cr on Ga substitutional site)



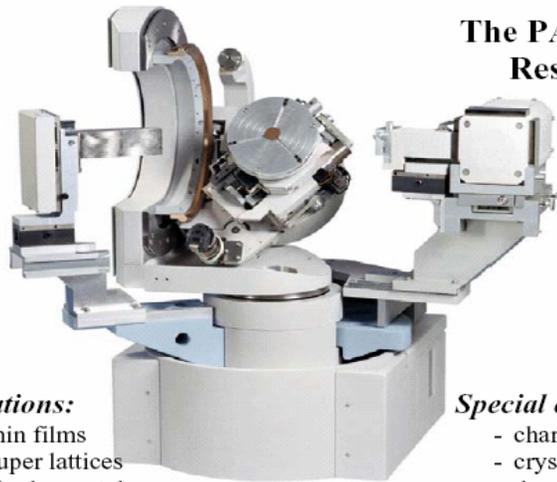
Uniform Cr distribution



Cr segregation in sample grown at 825°C



## **NEW addition to LE-CSSS September 1<sup>st</sup>** **New High resolution X-Ray Diffractometer**



### **The PANalytical X'Pert Pro Materials Research X-ray Diffractometer.**

*Diffractometer for thin films, single  
crystals and powders from room  
temperature up to 900°C using  
CuK $\alpha$  radiations*

#### ***Applications:***

- thin films
- super lattices
- single crystals
- polycrystalline samples
- powders and amorphous materials
- air sensitive materials

#### ***Special capabilities:***

- characterize primary and secondary phases
- crystal orientation
- degree of crystallinity
- stresses in the crystal structure
- spatial mapping (100 x 100 mm motion) of sample surfaces
- high speed wide angle x-ray diffraction and fast mode reciprocal space mapping

#### ***Thin film characterization:***

- characterize epitaxial films and film stacks
- layer composition, relaxation, thickness, curvature, mismatch, mosaicity
- degree of off cut
- super lattice period
- x-ray reflectivity on layered structures (crystalline and amorphous)

#### ***Some technical details:***

- 7 axis horizontal goniometer (Omega, 2Theta, Psi (Chi), Phi, X, Y, Z)
- flip focus Cu anode x-ray tube (line or point focus)
- high resolution: 5 arc sec in point focus mode / 19 arc sec in line focus mode
- low resolution 0.04 ° (Bragg Brentano slit optics)

#### ***Accessories:***

- three sample stages:
  - o standard 4" wafer mount
  - o solid sample holder (also used for powders)
  - o Anton Paar DHS 900 domed hot stage for data collection from RT up to 900 °C
- Incident beam optics
  - o 5 arc sec Ge(440) point focus module (Bartels monochromator)
  - o 19 arc sec hybrid (combination x-ray mirror + channel cut Ge(220) monochromator) line focus module
  - o manual divergence slit line focus module
- diffracted beam optics
  - o rocking curve receiving slit used with sealed Xe proportional detectors
  - o 12 arc sec Ge(220) monochromator used with sealed Xe proportional detectors
  - o manual anti scatter assembly used with X'Celerator high speed solid state linear array position sensitive detector

-ICDD PDF-2 XRD Database (2006-2007 version)

Contact Emmanuel Soignard (965 7242, [emmanuel.soignard@asu.edu](mailto:emmanuel.soignard@asu.edu)) for more information.

**NEW addition to LE-CSSS, February 2008**  
**Quantum Design MPMS SQUID magnetometer**  
**& electrical test system, 0-5 T, 2 -400 K**



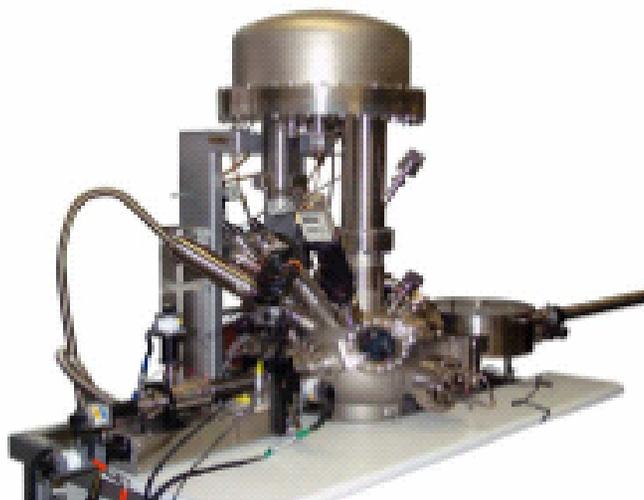
- SQUID AC Susceptibility Measurement (\*)  
- 0.1 Hz to 1KHz
- sensitivity:  $2 \times 10^{-8}$  emu at 0 T
- Ultra-Low Field Capability (\*)  $\pm 0.05$  G
- Reciprocating Sample Option (RSO) - DC Magnetization absolute
- sensitivity:  $1 \times 10^{-8}$  emu @ 2,500 Oe
- Continuous Low Temperature Control/Temperature Sweep Mode (CLTC) -
- Sweep rate: 0.001 - 10 K/min.

***Applications:***

- Magnetic materials
- Superconducting materials
- Paramagnetic centers in semiconductors and dielectrics

Contact Nate Newman (727 6934, [nathan.newman@asu.edu](mailto:nathan.newman@asu.edu)) for more information.

## New addition to LE-CSSS VG ESCALAB 220i-XL



The LeRoy Eyring Center for Solid State Science announces the availability of the VG ESCALAB 220i-XL. (shown in its current location in Goldwater B10) (donated to ASU by Intel Corp.)

It is expected to be fully operational/available for use on Sept. 1, 2007

### *Features:*

- Standard XPS analysis.
- X-Ray Photoelectron imaging.
- SEM-style imaging.
- Video imaging.
- Small spot analysis: 100 micron, 50 micron, and 20 micron.
- XPS Depth-profiling in small spot mode.
- XPS Angle-Resolved analysis.

### *X-Ray sources:*

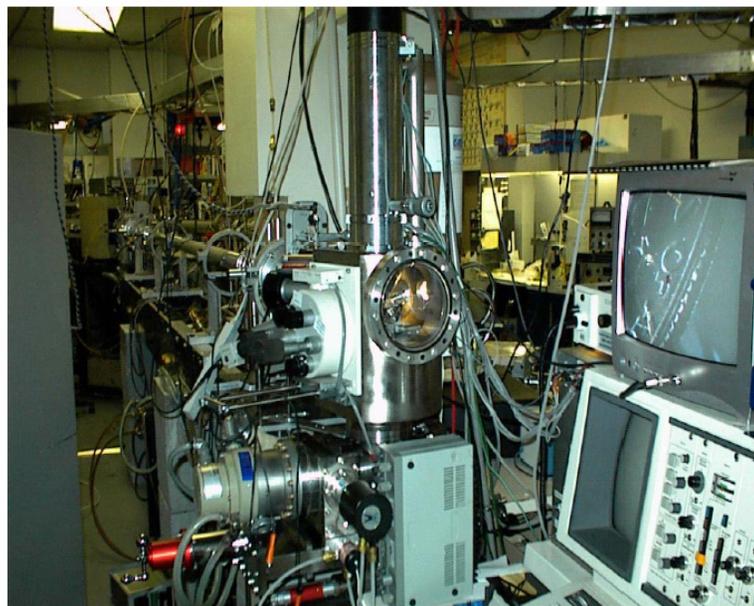
- Monochromated Al K-alpha. Linewidth 0.8 eV.
- Twin anode Al/Mg K-alpha

### *Sample handling:*

- Standard multiple sample mount bar.
- 4" diameter and smaller wafer mount platform with rotation.
- Sample stage x-, y-, and z-motion.

Please contact Tim Karcher at [Tim.Karcher@asu.edu](mailto:Tim.Karcher@asu.edu); GW B83; (480) 965-9070

## Nuclear Microprobe Analysis (Small Spot RBS and PIXE Analysis)

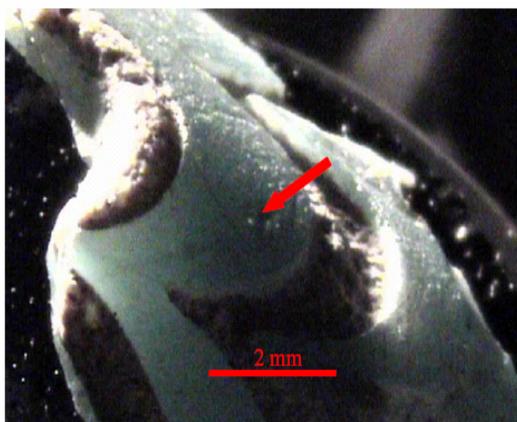


The capability of high spatial resolution PIXE and RBS analysis has been added to the IBeAM facility with the addition of a new beamline and focusing lens which provides a beam spot 100 – 500 X's smaller than the current 1 mm<sup>2</sup> spot size. The sample chamber incorporates an SEM (scanning electron microscope) and an optical microscope (with CCD camera) for small sample viewing and beam localization. Our anticipation is that the Nuclear Microprobe will find useful applications in a number of areas where improved spatial resolution in elemental analysis is

required. Some examples would include: Geology (mapping grains and crystals), Physics and EE (elemental analysis of microstructures and devices) and Life Sciences (elemental mapping of cells).

Optical image of a piece of ancient Egyptian decorated glass. Red arrow at left indicates location of 20 micron dia. proton beamspot.

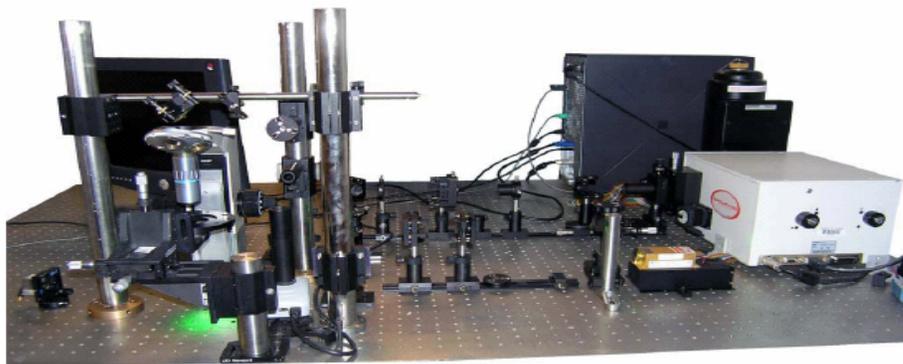
It is fully operational and currently available for use.



Contact Barry Wilkens ([barry.wilkens@asu.edu](mailto:barry.wilkens@asu.edu)) for more information.

## **New addition to LE-CSSS Raman spectroscopy facility**

**Instrument: custom System w/ 532 nm excitation wavelength  
Currently available in Physical Science C37 (PSC 37)**



*State of the art custom-built notch filter based high throughput Raman system with an excitation wavelength of 532 nm.*

- Maximum available power: 100 mW
- Special resolution: < 5 microns
- PI Liquid nitrogen cooled detector and Acton spectrometer
- Can collect down to  $\sim 100 \text{ cm}^{-1}$ .

*No or minimal sample preparation*

- NON DESTRUCTIVE
- Works for most liquid, gas or solid
- pure phase, multi phases, glasses, single crystals... and even artwork such as painting...

*Some applications*

- phase identification (compare to a published spectrum/ standard)
- investigate small chemical/structural
- strain/defects in some material
- sample mapping
- *in situ* characterization at high pressure and or temperature or within a medium

*Some limitations:*

- most metals are difficult / impossible to observe
- sample fluorescent
- symmetry of the material

Contact Emmanuel Soignard (965 7242, [emmanuel.soignard@asu.edu](mailto:emmanuel.soignard@asu.edu)) for more information.

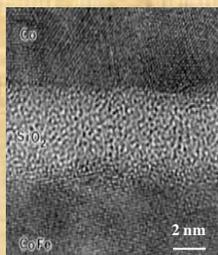
The purpose of the Center for High Resolution for Electron Microscopy is to promote the application of electron microscopy methods to problems of current scientific and technological importance.

The Center is a regional resource for applications involving imaging, microanalysis, electron diffraction, electron holography, and surface microscopy, as well as developments in methods and instrumentation.

Specific aims of the Center include:

- to provide access to advanced research tools;
- to provide advice on state-of-the-art techniques;
- to offer educational opportunities such as advanced schools and research workshops.

### Transmission Electron Microscopy



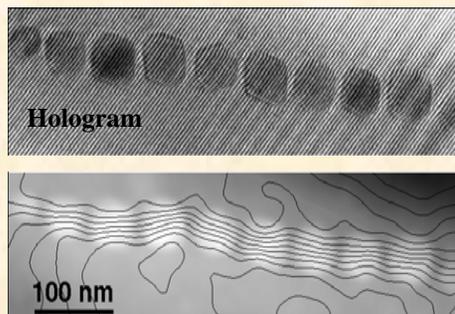
Cross-sectional TEM image revealing microstructure of Co/SiO<sub>2</sub>/CoFe magnetic tunnel junction [1].

### High-Resolution Electron Microscopy

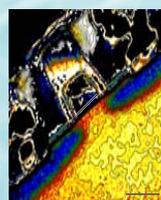


Cross-sectional electron micrograph showing misfit dislocations at sapphire/AlN interface.

### Electron Holography

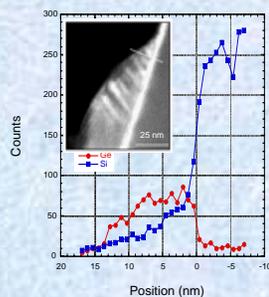


Two-dimensional imaging of magnetic field associated with tiny magnetite crystals in magnetotactic bacteria [2].



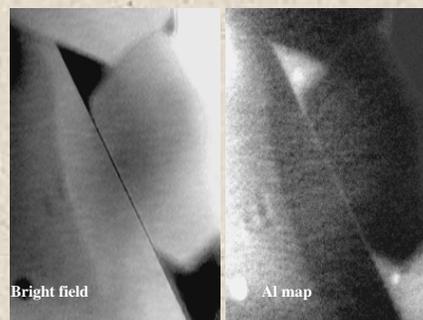
Electrostatic potential within 0.13 micron transistor device quantified using electron holography [9].

### Scanning Transmission E.M.



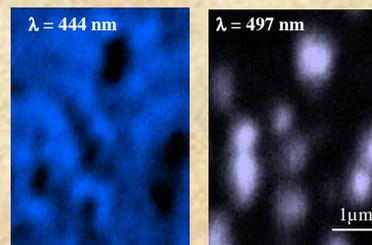
Energy-dispersive X-ray analysis with sub-nm STEM probe reveals Si interdiffusion into epitaxial Ge island [3].

### Energy Filtered Imaging



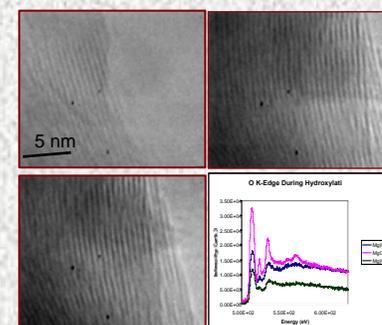
Grain boundary solute distribution in Si<sub>3</sub>N<sub>4</sub>/SiC ceramic densified with Y<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> sintering aid [4].

### Cathodoluminescence



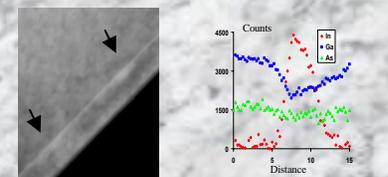
Plan-view CL images from InGaN used to correlate structural defects with light emission [5].

### Environmental Cell Microscopy



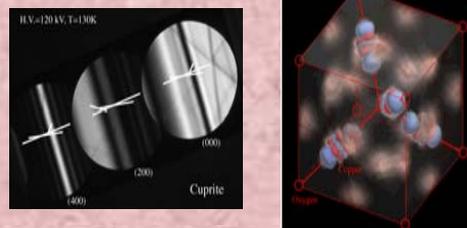
Progressive hydroxylation of MgO nanocrystal with associated change in EELS O<sub>K</sub>-edge [7].

### NanoSpectroscopy

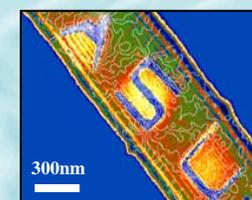


Cross-sectional image of InGaAs quantum dots in GaAs matrix and EELS elemental profile [8].

### Convergent Beam Electron Diffraction



Quantitative CBED pattern yields charge density map showing d-orbitals in cuprite crystal [6].



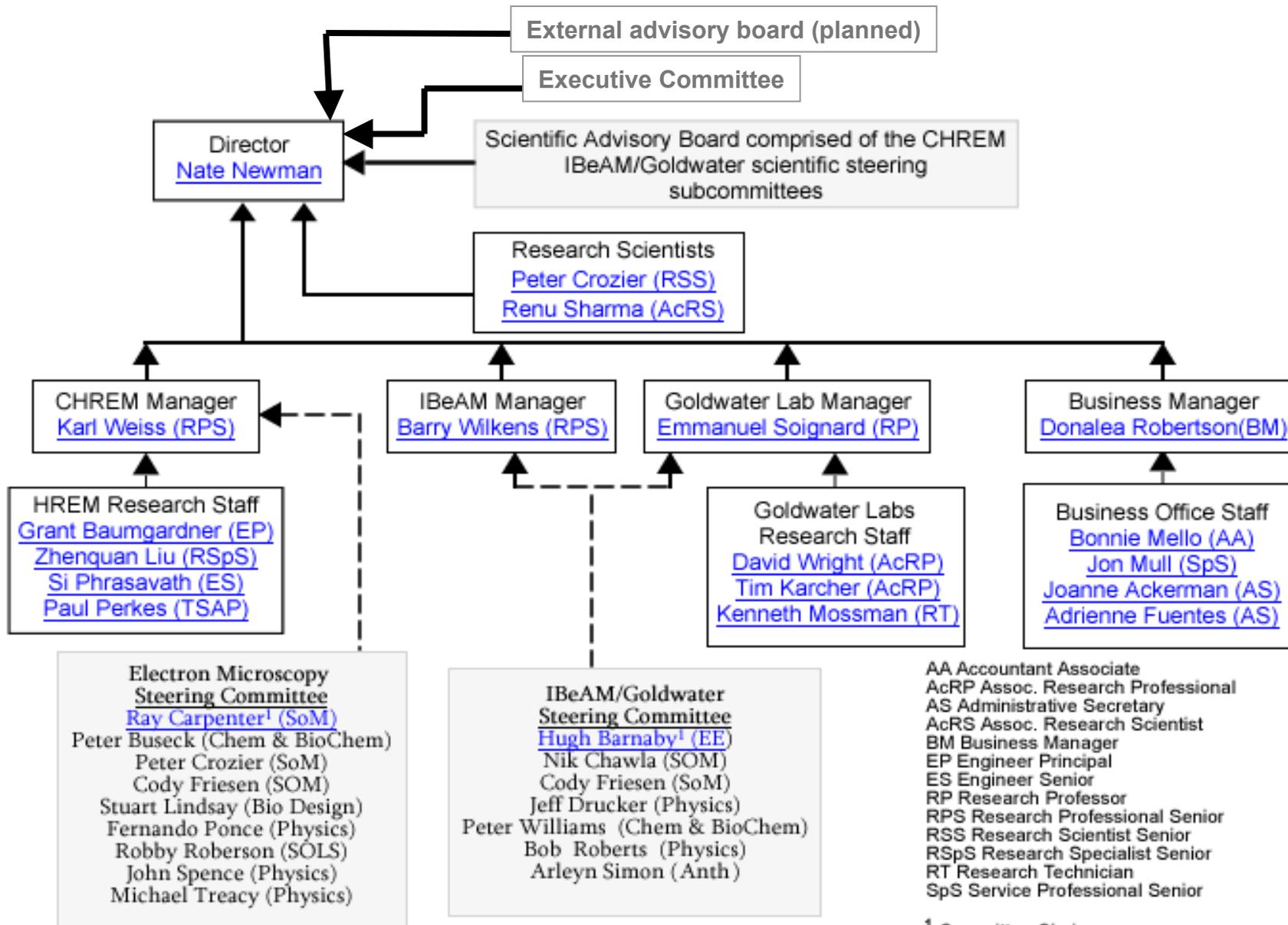
URL: <http://www.asu.edu/clas/csss/chrem>

#### References

- [1] D. J. Smith, et al., J. Appl. Phys., 83 (1998) 5154.
- [2] R. E. Dunin-Borkowski, et al., Science, 282 (1998) 1868.
- [3] S. A. Chaparro, et al., Phys. Rev. Lett., 83 (1999) 1199.
- [4] K. Das Chowdhury, et al., J. Amer. Cer. Soc., 78 (1995) 2579.
- [5] S. Srinivasan, et al., Appl. Phys. Lett., 2001 (in press)
- [6] J. M. Zuo, et al., Nature, 301 (1999) 49.
- [7] M. J. McKelvy, et al., Chem. Mater., 13 (2001) 921.
- [8] P. A. Crozier, et al., Appl. Phys. Lett., 2001 (in press)
- [9] M. A. Gribelyuk, et al., Phys. Rev. Lett., 2001 (in press)

## Major Instrument Acquisitions

<b>1970</b> JEOL 100B2	<b>1995</b> PHILIPS CM-200*
<b>1974</b> JEOL 100B4	<b>1996</b> LEO 912*
<b>1979</b> VG HB-5	<b>1997</b> JEOL 840*
<b>1980</b> PHILIPS 400T	<b>1997</b> VG HB-501
<b>1980</b> JEOL 200CX	<b>2000</b> JEOL JSM 6300
<b>1981</b> PHILIPS 400T FEG*	<b>2001</b> JEOL 2010F FASTEM*
<b>1984</b> JEOL 4000 EX*	<b>2002</b> TECNAI F20*
<b>1986</b> PHILLIPS EM 300	<b>2003</b> XL 30 ESEM FEG*
<b>1988</b> PHILIPS 430	<b>2004</b> Nova Nanolab FIB*
<b>1989</b> JEOL 2000FX*	<b>2006</b> VG-220i imaging XPS
<b>1989</b> TOPCON 002B*	<b>2007</b> Panalytic high-res. X-ray diffractometer
<b>1990</b> VG HB-501	<b>2007</b> Quantum Design SQUID Magnetometer
<b>1992</b> RBS I-beam accelerator	
<b>1993</b> AES Physical Electronics 590	
<b>1993</b> XPS Kratos XSAM 800	



## **Regional Aberration Corrected Microscopy Center (Ray Carpenter, chair)**

### **Observations:**

- 1. no aberration corrected electron microscopes in Southwest.**
- 2. ASU is best qualified university in the US to construct and operate a regional center.**

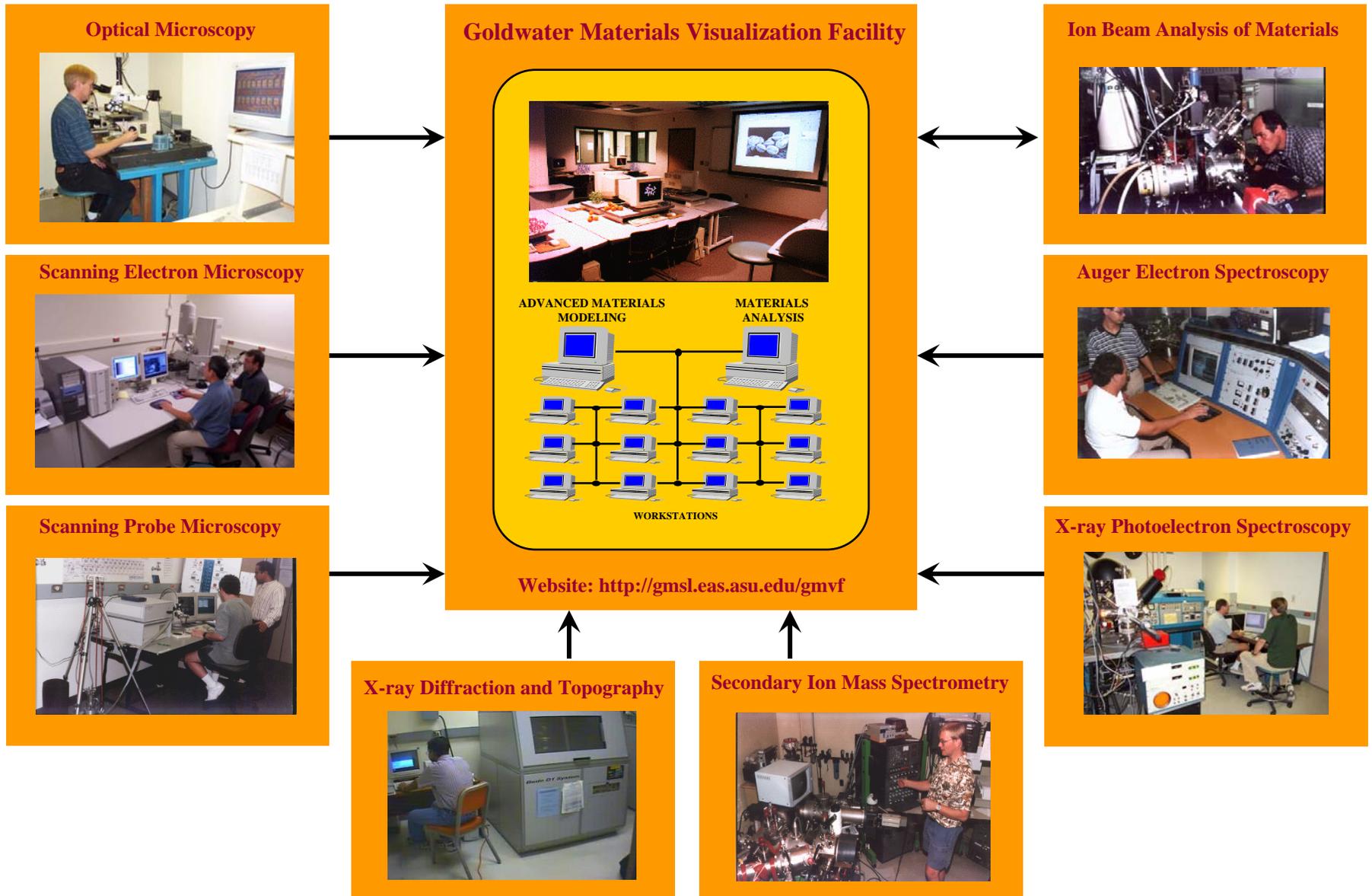
### **1. Personnel.**

- 2 tenure track materials science/electron microscopy in SOM**
- 2 permanent microscope researchers/operators**
- 2 postdocs**
- 1 computer support staff**
- 2 microscope maintenance staff**
- 1 center management staff**

### **3. Equipment**

- aberration corrected STEM capable microscope**
  - optimized for chemically sensitive imaging, electron and x-ray emission nanospectroscopy;**
- aberration pre-and post-specimen corrected STEM (TEM/STEM)**
  - for chemically sensitive imaging, HREM imaging, tomographic imaging, holography and nanospectroscopy**
- TEM/STEM microscope with chromatic aberration corrector as well as spherical aberration corrector, cryostage, monochromator, and energy loss spectrometer**
  - optimized for biomaterials nanocharacterization**
- corrected microscope (TEM, TEM/STEM or DSTEM ) with nanospectroscopic capability and ultra fast detection systems**
  - for *in-situ* chemical reaction and phase transformation research.**

# INTEGRATED MATERIALS ANALYSIS & ADVANCED COMPUTATIONAL MODELING





## Science is Fun

- **Brings excitement of scientific discovery to K-12 classrooms via interactive, minds-on demonstrations & explorations**
- **100 schools, >20,000 students visited**
- **UNDERGRADUATE INTERNS / RESEARCH ASSISTANTS**
  - **enroll in 3-credit Science is Fun Service Learning Internship (UNI 484).**
  - **Trained & present science explorations at local schools throughout greater Phoenix metropolitan area.**
- **<http://www.asu.edu/clas/csss/scienceisfun/>**

# Industrial Associates Program for Center for Solid State Science

Director, Dr. Renu Sharma

Renu.sharma@asu.edu

480 965-4541

# Membership-Based Structure

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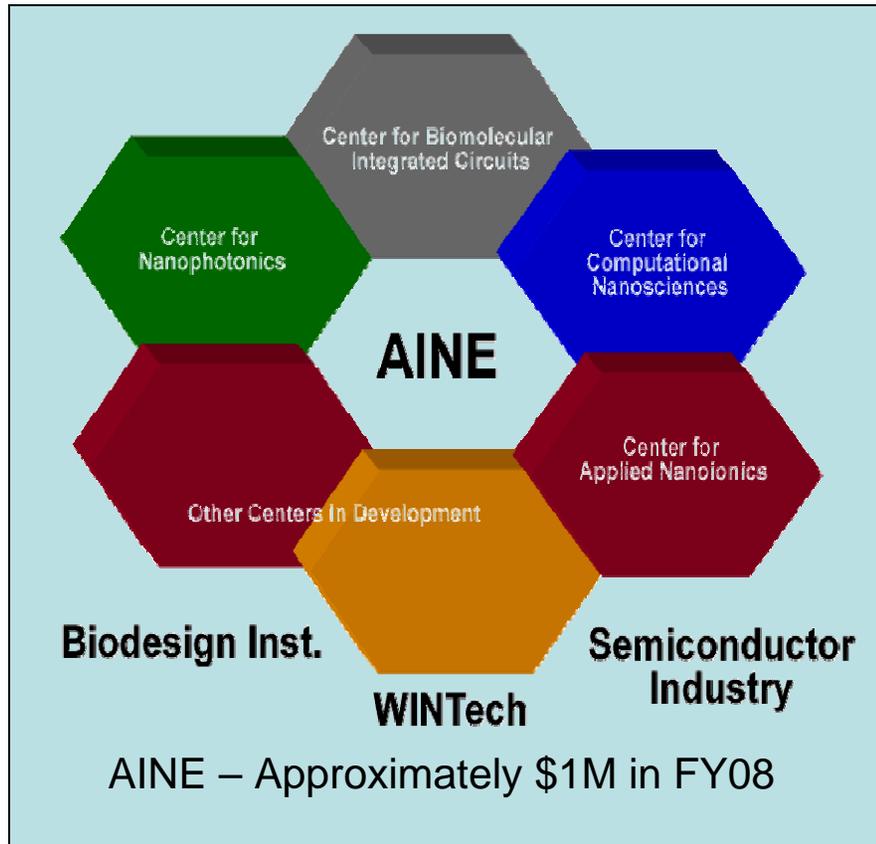
- Industrial Associates Program (IAP) established in 1988 and membership based.
- Members over the past 5 years include Intel, Motorola, Monsanto, Delphi, Chevron, Heraeus, IBM, Nanostellar, Exxon, Dow Chemical ( Union Carbide), Shell, Seagate, Ford, NanoTEM, HP, Agilent, AMD, Qynergy, Valence Technology.
- Membership Categories:

Platinum	\$40k/year
Gold	\$10k/year
Silver	Hourly quotes

# Aligning with major ASU initiatives, programs and projects

## School of Materials Research areas

- ✓ *Functional Materials*
- ✓ *Computational Materials Science*
- ✓ *Energy*
- ✓ *Electron microscopy*
- *Soft solids*



### Arizona Institute for Renewable Energy (AIRE)

Capabilities and Infrastructure

USE INSPIRED • TRANSDISCIPLINARY • INTELLECTUAL FUSION • SOCIAL EMBEDDEDNESS

**AIRE – Arizona Institute for Renewable Energy, ~\$800K in FY08 & MRSEC effort (van Schilfgaarde)**

### ASU's Flexible Display Center: Resolving U. S. Industry's Barriers to Scale-Up

The Gen II pilot line capable of demonstrating the manufacturing and scaling of large area, lightweight integrated photovoltaic devices on flexible substrates

USE INSPIRED • TRANSDISCIPLINARY • INTELLECTUAL FUSION • SOCIAL EMBEDDEDNESS

**Flexible Display Center- Multi-million dollar commitment per year in terms of 75% RID return and Building O&M/Debt Service**

*Integrated Materials- \$200K in support for CEMAPS in 08*