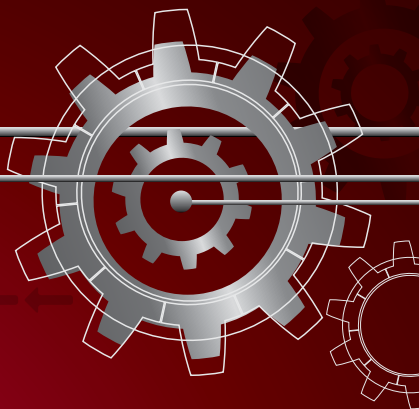


NCMN

Nebraska Center for Materials and Nanoscience

University of Nebraska–Lincoln

Central Facilities



www.unl.edu/ncmn



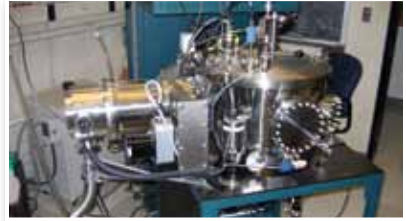
Cryogenics



Crystallography



Electron Microscopy



Materials Preparation



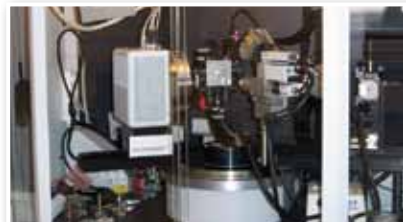
Metallurgical and
Mechanical Characterization



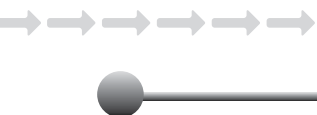
Nanofabrication



Scanning Probe Microscopy



X-Ray Materials Characterization



The Nebraska Center for Materials and Nanoscience

About the Center

The Nebraska Center for Materials and Nanoscience at the University of Nebraska–Lincoln strives to be a center of excellence in research, graduate and postdoctoral education and service in the area of materials science and engineering. NCMN is a multidisciplinary organization with over seventy faculty members in six major departments in the College of Arts and Sciences and Engineering that provides visibility and program strength in selected materials research areas. Major collaborative initiatives under way are an NSF/EPSCoR research cluster in nanostructured materials and a joint industry-government-university consortium in materials research for ultra-high-density information storage. NCMN is responsive to industrial efforts in the state that rely on materials analysis and processes for their economic growth.

Availability

These facilities are available to all UNL faculty as well as companies in Nebraska and elsewhere. Please contact the facility specialist to schedule equipment use or training.

About this Brochure

There are eight Central Facilities to support the NCMN's mission:

- Cryogenics
- Crystallography
- Electron Microscopy
- Materials Preparation
- Metallurgical and Mechanical Characterization
- Nanofabrication
- Scanning Probe Microscopy
- X-Ray Materials Characterization

The purpose of this brochure is to serve as a guide to the capabilities and equipment available in each facility. Facility phone numbers and e-mails are listed to answer additional questions.

Cryogenics

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Dr. Shah Valloppilly, Specialist

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www.unl.edu/ncmn/facilities/cryogenics.shtml

Cryogenics:

Continuous supply of liquid nitrogen and liquid helium for low temperature research, cold traps, etc.



Liquid Nitrogen and Liquid Helium Supplies



Liquid Nitrogen

A 230 liter supply of liquid nitrogen is maintained at this facility. Researchers can transfer LN2 to their own dewars (typically 1-30 L) as needed and are billed on a per liter basis.

Liquid Helium

LHe orders are delivered to UNL only once a week (Tuesdays), and orders must be submitted to the specialist by the previous Thursday to ensure that the request is placed in time. A delivery location (building and room #) should be specified. LHe can be ordered in 60 or 100 liter containers.

Emergency deliveries of LHe (available with ~ 2 days notice) are available, but are subject to a special freight charge. (This charge is about \$150-\$200)

Crystallography

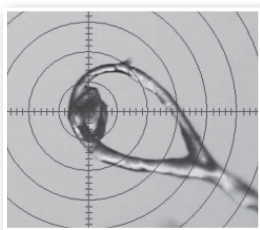
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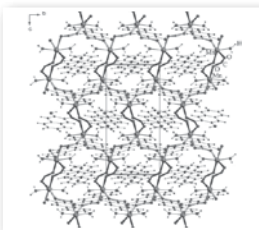
Crystallography:

On-site small molecule crystal structure determination and analysis including unit cell determinations, complete structure determinations, and collection of single crystal data.

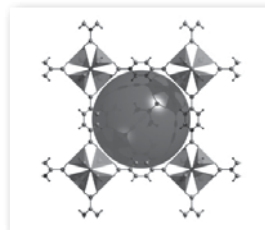
- Unit cell determinations
- Complete structure determinations
- Collection of single crystal data
- Bruker AXS SMART Apex instrument
- Leica ZOOM 2000 microscope
- Meiji stereo zoom microscope with polarizer attachment



Crystal on a Cryo-loop



Depictions of Crystal Structures



The Nebraska Center for Materials and Nanoscience (NCMN) Crystallography Central Facility has been established to bring on-site small molecule crystal structure determination and analysis to UNL. Services provided include unit cell determinations, complete structure determinations, and collection of single crystal data for users to solve and refine themselves. A Bruker AXS SMART APEX instrument equipped with an Oxford Cryosystems 700 series Cryostream is used to collect the single crystal data. A Meiji stereo zoom microscope with polarizer attachment is used to select single crystal samples. A special fitting for the Meiji microscope allows a digital camera to photograph the crystals.

The facility holds licenses for the Bruker AXS software, including SMART, SAINT, SADABS, SHELXTL and XSELL, as well as ATOMS and the Cambridge Structural Database (CSD). There are also many free-ware crystallography programs which are maintained on the facility computers, such as PLATON and ORTEP. There are one Mac and 3 PC's available in the facility for collecting data and performing structure determinations. The specialist is available daily for consultation and can provide advice on crystallization techniques or problems. The Bruker AXS SMART APEX CCD X-ray Diffractometer is equipped with a 4K Apex CCD detector, a Mo sealed-tube X-ray source, and MonoCap collimator, and an Oxford Cryosystems 700 Series Cryostream Cooler. The data collection temperature ranges from 80 to 400 K. The standard operating temperature is 120 K for materials that do not undergo phase transitions. Users are encouraged to communicate their preference for data collection temperature.

Facility Policies

Data collection and structure determinations are performed on a first-come, first-serve basis. If you are preparing a highly unstable and possibly fleeting material, make arrangements ahead of time with the specialist. When a structure is considered final and complete, a CD will be written containing all pertinent data and distributed to the user who submitted the crystal. If users develop a later need for different pictures or new analysis of interactions, they should see the specialist with the archived CD in hand. A folder will be saved on your archive CD called FINAL. All pertinent files (.cif, .res, tables, ortep, etc.) will be saved in this folder.

You are responsible for safekeeping your own released data.

If you need to meet with the specialist, please visit the facility for questions and information.

Electron Microscopy

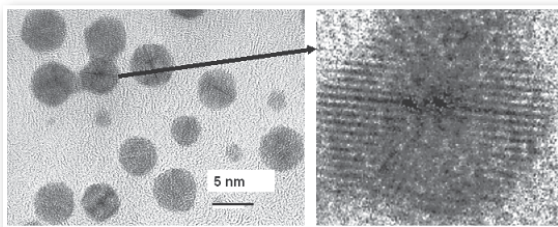
Professor Brian W. Robertson, Faculty Supervisor
Dr. Xingzhong “Jim” Li, Specialist

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Electron Microscopy:

Materials characterization of the topography, morphology, elemental composition, crystalline microstructure, crystal defects, and atomic arrangements of materials, largely on a scale from 10 micrometers down to the near-atomic level.

- Atomic-scale and submicron materials analysis
- 200 kV high-resolution electron microscope (JEOL JEM 2010 TEM)
- Analytical scanning transmission electron microscope (VG HB501 STEM)
- Well-equipped scanning electron microscope (JEOL JSM 840A SEM)
- Necessary support instruments



TEM images of FePt clusters
with facet feature

Enlarged cluster showing
lattice fringes

The function of the Central Facility for Electron Microscopy is to provide hands-on access to electron microscopes, sample preparation equipment plus data collection and data reduction instrumentation, along with advice, training and research collaboration. The scope of the facility is materials characterization of the topography, morphology, elemental composition, crystalline microstructure, crystal defects, and atomic arrangements of materials, largely on a scale from 10 micrometers down to the near-atomic level.

Main Equipment

The facility is equipped with:

1) JEOL JEM 2010 TEM

200kV, analytical/high-resolution mode, LaB6 filament, single-tilting and double-tilting sample holders. (Oxford EDS system, Gatan dual-view CCD camera, TSL texture analysis system, Digi-TEM beam control system.)

2) JEOL JSM 840A SEM

LaB6 filament, second electron and backscattered electron detectors, Kevex Quantum x-ray microanalyzer, digital imaging system.

3) VG Microscopes HB501 STEM

Field emission scanning transmission electron microscope, now including: Windowless x-ray microanalysis (Oxford/ 4pi), digital image acquisition (4pi), parallel-acquisition EELS, energy-filtered electron diffraction, differential phase contrast detector, electron beam induced nanofabrication facility.

Specimen Preparation Devices

- Emscope SC500 Au sputter coater
- Diamond blade slow speed saw
- SBT Model 910 lapping and polishing machine
- Branson 2200 ultrasonic cleaner
- VCR Group Inc. Dimpler D500i
- Gatan Inc. Model 691 PIPS
- Leica stereo microscope ZOOM 2000
- Intek optical microscope

Users of the Facility

The facility is accessible to all qualified researchers at UNL, at other universities, and in industrial and other laboratories on payment of the appropriate charges.

Under normal circumstance, these researchers will be expected to have the requisite background knowledge for electron characterization of materials before some training will be given for a particular instrument.

In some cases, research collaboration is possible for common research interests and/or if researchers need the capabilities of the equipment and experienced examination of samples but are unable to use the facilities for themselves. Limited full-service handling and examination of materials is available for these researchers according to availability of the facility specialist.

Materials Preparation

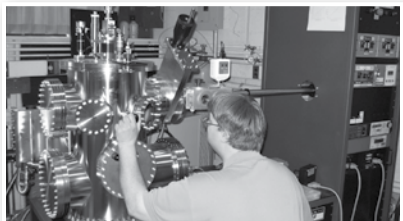
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Materials Preparation:

State-of-the-art equipment to prepare novel nanostructured thin films from a variety of materials.

- Two sputtering systems (3 guns each) to fabricate a variety of thin films, especially nanostructured films including overlayers, multilayers, granular solids, clusters, etc.
- E-beam Evaporator
- Two tube furnaces (Lindberg 54233 and Lindberg 55332)
- Vacuum pump stations for sample or target annealing, doping, and sintering



Preparation of epitaxial thin films using
AJA MBE system

Sputtering Systems

Two sputtering systems have been established to fabricate a variety of thin films, especially the nanostructured films including overlayers, multilayers, granular solids, clusters, etc.

Each sputtering system has three guns: two dc guns for conductive targets and one rf gun for conductive or nonconductive targets. The system is computerized to control the positions of the deposited substrates and twelve samples can be prepared in one vacuum run to avoid undesired changes of preparation conditions for one series of samples. The base pressure is about $1\sim 3 \times 10^{-7}$ Torr, and the preparation conditions, such as Ar pressure during sputtering and the sputtering rates of the targets, can be adjusted by controlling the Ar flow, sputtering power, etc.

Currently there are two gas channels, which may be expanded to four channels, and chemical gas reaction sputtering can be performed. The substrate temperature can be varied from room temperature to about 700°C. Each sputtering system also has one evaporator which can be used to evaporate thin films or fine particles.

E-beam Evaporator

The facility also houses an E-beam evaporator recently purchased from AJA international. The E-beam system is very similar in design to the sputtering system. The system has the following features:

- Four material pockets for deposition of single and multi layers and alloys.
- Can reach a base pressure $\sim 5 \times 10^{-9}$ Torr
- Can handle 4-inch substrates which can be rotated and heated up to 850°C
- A load lock chamber for quick removal and insertion of samples.

Heat Treatment Ovens

Two tube furnaces (Lindberg 54233 and Lindberg 55332) together with vacuum pump stations are available for sample (or target) annealing, doping, and sintering. The Lindberg 54233 oven has an operating temperature of $T_{\max} = 1500^{\circ}\text{C}$ and a working tube of diameter 2". The Lindberg 55322 oven has an operation temperature of $T_{\max} = 1200^{\circ}\text{C}$ and a working tube of diameter 2.5". Both ovens have programmed power supplies and the base pressure of the pump stations are about 2×10^{-6} Torr.

Metallurgical and Mechanical Characterization

Professor Jeff Shield, Faculty Supervisor
Dr. T.V. Jayaraman, Specialist

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www.unl.edu/ncmn/mech_charact

Metallurgical and Mechanical Characterization:

A large variety of equipment to characterize the mechanical and physical properties of materials including the failure analyses on components.

- Leica DM 2500 M Optical Microscope
- Olympus microscopes
- Rockwell, Knoop, and Vickers hardness testers
- Tension/compression testing machines (MTS, Instron, Satec, Tinius-Olson)
- Torsion testing equipment
- Other equipment: hot presses, cold-mounting facility, belt grinding, hand lapping, and fine polishing stations, and a variety of saws



Leica DM 2500 M Optical Microscope

This facility contains a large variety of equipment to characterize the mechanical and physical properties of a wide range of materials. The facility focuses on using the many materials characterization aspects to perform failure analyses on components. Samples can be analyzed to determine the failure mode and solutions to prevent further problems. Samples can also be prepared for the Electron Microscopy or X-ray Diffraction Facilities, if necessary.

Metallography utilizes various types of equipment to prepare and examine the microstructure of all types of materials. Available equipment includes a large fluid-cooled band saw, a water-cooled abrasive cut-off saw, and a diamond sectioning saw for sample sectioning; two hot presses for use with bakelite-type materials for mounting of samples, as well as facilities for cold-mounting of materials; a belt grinding station and two hand lapping stations for rough and coarse grinding; four variable speed stations for use with aluminum oxide and diamond compound for fine polishing. A Leica DM 2500 M Optical Microscope, which includes differential interference contrast capabilities, is available for sample viewing and image analysis capabilities. Photographs may be taken as computer image, 35mm, or via thermal image printing.

Mechanical testing facilities include hardness testing equipment such as regular and superficial Rockwell, Knoop, and Vickers testers. Other testing facilities include a MTS 20,000 lb. servo-hydraulic material testing system, an Instron 10,000 lb. tensile testing machine, Satec 100,000 and 400,000 lb. machines, and Tinius-Olson instrumented Charpy impact and torsion testers.

Metallography is the art and science of preparing materials for examination in the microscope. The metallography lab contains equipment to section, mount, polish, and etch materials to enable the user to observe the microstructure of the material of interest, which can then be observed using light or electrons as the imaging method. Mechanical testing utilizes various equipment, which is usually designed to physically pull a material to failure. This may involve static or slowly applied loads such as are obtained with the presses, or a dynamic load such as is obtained with the Charpy impact testing machine. Both machines record load during testing in order to measure and determine the strength parameters of the material. Hardness testing involves using a known load and a known indenter shape which is then pressed into a material. The depth of penetration of the indenter is proportional to the hardness of the material. Hardness data can be used to infer other mechanical properties of the material. The hardness testers available include both macrohardness and microhardness testers which produce indentations which are fairly large (.20" in diameter) to extremely small (a thousandth of an inch).

Nanofabrication

Professor Ned Ianno, Faculty Supervisor
Lanping Yue, Specialist

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www.unl.edu/ncmn/facilities/nanofab.shtml

Nanofabrication:

Fabrication of electronic, magnetic and other nanostructures.

- Zeiss field-emission icroscope
- Raith e-beam lithography
- Focused ion-beam workstation
- Suss mask aligner
- Trion reactive ion etcher
- AJA e-beam evaporation system, ovens, and others



FEI Strata 200xp focused ion beam (FIB) system

Focused-Ion Beam Workstation

FEI Strata 200xp focused ion beam (FIB) system provides strong capability and tremendous flexibility of nanofabrication to majority of materials. The Ga^+ ion beam scans through a desired location and mills a very small area around the region of interest. It is also a precise instrument that can be applied to the fabrication of microelectronic and optoelectronic devices, such as direct-write lithography, circuit modification, failure analysis, defect characterization, and ion beam assisted etching and micromachining.

This system is an advanced tool for rapid, precise micromachining and deposition of submicron features in various substrates and materials. It is also a superb general purpose tool for SEM imaging, grain structure analysis, high aspect ratio probe milling, fabrication of nano-patterns, and other related applications. Two gas injection systems (GIS) are equipped. Pt GIS is used for deposition of conductive and protective platinum layer; selective carbon mill GIS is for enhanced etch of C-containing samples such as polymers and other resistive materials. Other functions include an in-situ optical microscope, real time milling monitor, and auto FIB programmable milling.

- Accelerating energy range between 5-30 keV
- Beam current range from 1 pA-11.5 nA for 10 apertures
- Beam diameter is 8-500 nm
- Less than 20 nm minimum resolution in milling and patterning

Electron Beam Lithography System

This lithography instrument integrated with the Zeiss field-emission electron microscope to form a complete electron beam lithography system. A new Zeiss Supra 40 FESEM allows imaging of sample surfaces with very high resolution. Ultimate resolution is below 1.2 nm at high energies and a few nm at sub keV energies for ultimate analytical work on nano-particles and polymer materials prone to electron beam damage.

- Energy of electrons: 200 V-30 kV
- Resolution to 1.3nm @ 15kV; 2.1 nm @1kV
- Working distance: 2-30 mm

Reactive-Ion Etching System

A recently acquired RIE system is a plasma etch system with state-of-the-art plasma etch capability using single wafers, dies or parts. The RIE system has up to seven process gases (CF_4 , O_2 , Ar, He, Cl_2 , BCl_3 , HBr) which are used to etch films such as silicon oxide, silicon nitride, polysilicon, aluminum, GaAs and many others. This reactor can also be used to strip photoresist and other organic materials.

Samples are loaded into the process chamber via the vacuum load lock. This feature increases user safety by preventing contact with the process chamber and any residual etch by-products. The load lock also allows the chamber to remain permanently under vacuum thereby keeping out moisture and keeping the reaction chamber free of possible corrosion.

Scanning Probe Microscopy

Professor Sy-Hwang Liou, Faculty Supervisor
Dr. Lanping Yue, Specialist

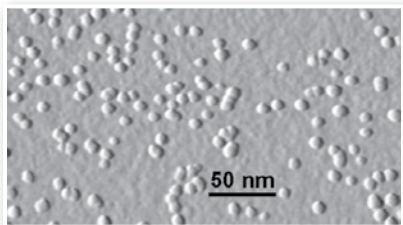
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Scanning Probe Microscopy:

Nanometer-scale characterization of materials by using:

- Scanning Tunneling Microscopy (STM)
- Atomic Force Microscopy (AFM)
- Magnetic Force Microscopy (MFM)

NCMN Scanning Probe Microscopy (SPM) Facility provides nanometer-scale characterization of materials by using Scanning Tunneling Microscopy (STM), Atomic Force Microscopy (AFM), and Magnetic Force Microscopy (MFM), etc. SPM can provide a wealth of information from topography, surface morphology, to magnetic phase or friction analysis, including line width, grain size, pitch and depth, roughness measurements, sectioning of surfaces, power spectral density, particle analysis, surface defects, and pattern recognition, etc.



AFM image of monodispersed, sub-10nm Fe clusters produced by inert gas condensation

Equipment

The facility is equipped with:

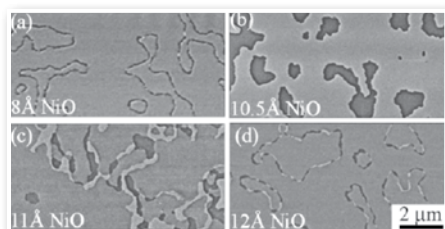
1) EnviroScope Atomic Force Microscope (ESCOPE)

The new Digital Instruments ESCOPE combines AFM imaging with environmental controls and hermetically sealed sample chamber to perform contact, lateral force, and TappingMode atomic force microscopy in air, vacuum, or a purged gas, as well as a heating environment. With advanced environmental capabilities, users can observe sample reactions to a variety of complex environmental conditions while scanning. In addition, the new system supports STM in air or vacuum.

2) Dimension 3100 SPM system

The Digital Instruments Nanoscope IIIa Dimension 3100 SPM system is a multifunction scanning probe microscope to measure surface characteristics for a large variety of materials, such as nanoparticles, polymers, DNA, semiconductor thin films, magnetic media, optics and other advanced nanostructures.

Our MFM facility can scan samples in external magnetic fields, which is useful for magnetic domain imaging. The magnetic fields available using permanent magnets are ± 0.25 T perpendicular to the sample and ± 0.45 T parallel to the sample. The resolution of MFM tips modified by Focused-Ion-Beam (FIB) milling is around 15 nm, which is state-of-the-art capability currently.



MFM domain images of Co/Pt multilayers with 8, 10.5, 11 and 12 Å NiO interlayers.

The facility is available to all qualified researchers, who are properly trained at UNL, on payment of the appropriate charges for equipment use. Research collaborations are welcome from all UNL faculty as well as companies in Nebraska and elsewhere.

X-Ray Materials Characterization

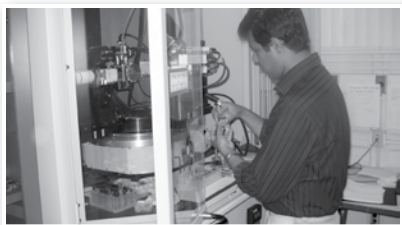
Professor Roger Kirby, Faculty Supervisor
Dr. Shah Valloppilly, Specialist

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www.unl.edu/ncmn/xray

X-Ray Materials Characterization:

Materials identification and characterization through non-destructive, X-Ray Diffraction (XRD) and/or X-Ray Fluorescence (XRF) techniques.

- Bruker-AXS D8 Discover X-Ray Diffractometer with GADDS Area Detector
- Rigaku D/Max-B diffractometer – large-angle and small-angle goniometers



Bruker-D8 Diffractometer in
Reflectometry set-up

The NCMN X-Ray Characterization Facility is dedicated to materials identification and characterization through non-destructive, X-Ray Diffraction (XRD) and/or X-Ray Fluorescence (XRF) techniques. The following equipment is utilized: 1) Bruker-AXS D8 Discover High-Resolution Diffractometer; 2) Rigaku D-Max/B Horizontal Q/2Q X-Ray

Diffractometer (with normal and small angle goniometer); 3) Rigaku X-Ray Diffraction Laue Camera System (transmission and back reflection).

Bruker-AXS D8 Discover High-Resolution Diffractometer

This recently acquired system is a state-of-the-art machine including HI-STAR area detector, centric 1/4-circle Eulerian cradle, domed hot stage, hi-flux in-plane hardware, laser/video sample-alignment system, Göbel mirror, V-groove Ge crystal monochromator, fine tilt stage, and dual-beam path analyzer module. The system can be configured for grazing-incidence in-plane XRD, grazing-incidence XRD, x-ray reflectivity, high-temperature XRD, high-resolution XRD (rocking curves, reciprocal space maps), texture (pole figures), residual stress, and microdiffraction and capillary diffraction.

Rigaku D/Max-B Diffractometer

X-Rays are produced by a 2 kW, copper target, sealed tube or diffracted beam then converges (is “focused”) into a diffracted beam monochromator which removes all radiation except the Cu K α wavelength (about 1.544 Å) which then enters a scintillation counter. The sample and detector are rotated with respect to the incident beam at angles θ and 2θ , respectively. A typical XRD scan consists of a plot of detector angle (2θ) vs. diffracted intensity. These diffractograms can be printed out or saved to disk in application-friendly ASCII format. Analysis can be carried out in the facility on any of three PCs equipped with qualitative and quantitative XRD software and access to the latest ICDD powder diffraction file database.

Most powder, thin-film, and bulk samples are acceptable. The sample holders can accommodate samples up to 30 x 30 x 10 mm in size (sometimes larger) with the 30 x 30 mm surface tangent to the diffracting circle. The amount of material needed for a diffraction pattern depends on the diffracting power of the sample and the type of analysis desired. For example, phase identification of a well crystallized powder, with small (< 40 nm) particle size, may only require 1 mg. or less.

Rigaku X-Ray Diffraction Laue Camera System

This instrument consists of a 2 kW, copper target x-ray tube, a Polaroid XR-7 camera system, a manual goniometer with three rotational axes, and a movable platform. The camera uses film that is sensitive to x-radiation. The Laue x-ray diffraction method requires a continuous (white) spectrum of x-ray which is what you get from an unfiltered, unmonochromatized x-ray tube. The single crystal sample is fixed in position throughout the x-ray exposure with respect to the incident beam and the camera. Using this configuration, we obtain diffraction peaks (show as white spots on film) from many sets of crystal planes simultaneously because, although the geometry and d-spacing are different for each set of diffracting planes, there is a wavelength of radiation present for which the Bragg law is satisfied. The relative location of these spots on the film can be used to determine crystal structure and orientation. The system can be set up to allow transmission (sample between incident radiation and film) or back-reflection (film between sample and incident radiation Laue patterns).

Useful Information for NCMN Facility Users

Charge Policy

Each NCMN facility assesses charges for equipment use. Charges are variable and are assessed to offset the costs of running each facility and to ensure the use of the facility by qualified researchers. These rates are set at the beginning of the fiscal year based on the previous year's operating costs and expenses.

Most facilities charge for equipment use by the hour with rates depending on which equipment is used as well as whether the specialist is used. Those who wish to use the equipment without specialist assistance must contact the facility specialist for training. *(Please note: this option is not available for users outside UNL.)*

Charge Rates

Charges are subject to yearly revision and are different for each facility. Please contact the NCMN business office or the facility personnel to obtain the latest prices.

For More Information

For specific equipment-related questions about each facility, contact the facility personnel. For general questions about NCMN facilities, which charge rates apply to you, or any questions about billing, please contact the NCMN Business Manager's Office at 402.472.6072.

Specialized Research Facilities

State-of-the-art equipment: 14 Tesla NMR spectrometer for solid-state NMR, NIMA Langmuir-Blodgett Trough for monolayer and multilayer films, atomic force microscopes, high-field superconductive solenoids, a SQUID magnetometer, angle-integrated photoemission and electron spin analysis facilities, Raman and Brillouin laser light-scattering facilities, comprehensive laboratory for the study of magnetic materials, high-T_c superconducting materials, photoemission and inverse photoemission spectrometers including spin-polarized inverse photoemission, and dedicated minicomputers for theoretical calculations, pulsed laser facilities, atomic force microscope, and comprehensive ellipsometer laboratory.





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