

CINT Jump Start User Capabilities

Synthesis Capabilities

— **Thin Film and Epitaxial Growth**

- III-V Semiconductor molecular beam epitaxy
- Metal-organic chemical vapor deposition
- Group IV molecular beam epitaxy
- Metallic multilayer synthesis
- Energetic neutral atom beam lithography

— **Nano/Micro Fabrication**

- Electron beam lithography
- Photolithography
- Patterned metal and dielectric thin films
- Plasma etch and ion milling
- (also see MicroElectroMechanical Systems)

— **Nanomaterials Synthesis**

- Polymeric monolayer systems
- Photo-catalytic growth of nano-assemblies
- Mesoporous materials
- Colloidal assembly/Surface templating
- Oxide nanomaterials
- Colloidal synthesis: Semiconductor, metal and magnetic nanocrystals
- Chemical synthesis of nanoscale electronic materials
- Thin-film preparation
- Intermetallic and oxide synthesis and crystal growth
- Organic synthesis and crystal growth
- Polyoxoniobate chemistry

— **Biomaterial Synthesis**

- Lipid membranes and self-assembled films
- Bio-inspired and bio-compatible materials
- Biochemical techniques and instrumentation
- Ligand development

Characterization Capabilities

— **Scanning Probe Microscopy**

- Atomic force microscopy, near-field and fluorescence imaging
- Atom tracking scanning probe microscopy
- Interfacial force microscopy
- (Also see Scanning Probe Microscopy Facility)

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___ **Optical Microscopy/Spectroscopy**

- Optical spectroscopy
- Optical microscopy
- Ultrafast laser spectroscopy

___ **Electron Microscopy**

- Low energy electron microscopy
- (Also see Electron Microscopy Laboratory)

___ **Nuclear Magnetic Resonance**

- Nuclear magnetic resonance of ceramics
- (Also see NMR Facility)

___ **Mechanical Properties**

- *In-situ* mechanical testing
- Mechanical properties from near-surface layers

___ **Transport**

- Physical characterization at extremes of parameter space
- Low temperature electronic transport

___ **Theory and Simulation**

Leveraged Facilities

___ **Ion Beam Materials Laboratory**

___ **MicroElectroMechanical Systems**

___ **Nuclear Magnetic Resonance Facility**

___ **Electron Microscopy Laboratory**

___ **Scanning Probe Microscopy Facility**

National User Facilities

___ **Los Alamos Neutron Scattering Center – LANSCE**

___ **National High Magnetic Field Laboratory – NHMFL**

___ **Combustion Research Facility – CRF**

CINT Jump Start User Capabilities

Thin Film and Epitaxial Growth

III-V Semiconductor molecular beam epitaxy

The molecular beam epitaxy (MBE) capabilities allow the growth of As-based III-V compound semiconductors. The system specializes in high-purity, high-mobility materials grown with monolayer precision. Due to the high-mobility nature of the system, only n-type doping using Si is available. Areas of interest for growth available to CINT Users during jump-start operations include:

- Low dimension semiconductor systems
- Quantum transport
- Electronic devices based on intrasubband transitions.

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Metal-organic chemical vapor deposition

The MOCVD capabilities will enable researchers to investigate the synthesis of novel materials and complex structures involving the use of III/V compound semiconductors. Recent advances in MOCVD growth techniques have enabled the preparation of quantum dot (QD) structures. These QDs can be formed through self-assembly, selective area growth, or patterned growth. Investigation of the properties of these materials holds the promise of enhanced device performance. We are exploring a variety of methods to control the size, shape, and density of the QDs and their resulting physical properties. The growth and materials science of GaN-based semiconductors has recently become one of the dominant areas of compound semiconductor research as well as MOCVD science. We are actively investigating all aspects of the growth and materials science and device design related to GaN-based semiconductors. This includes investigations into the details of defect physics of these materials, a detailed understanding of the mechanisms of dislocation formation, as well as the preparation of low dislocation density substrates. The capabilities that are available to CINT Users during jump-start operations include:

- MOCVD growth of a wide range of III-V semiconductors from InSb- and GaN-based materials to the more conventional GaAs
- Growth of quantum dots using self-assembly as well as selective area or patterned growth
- Investigations into the use of surfactant materials to enhance controlled growth of heterostructures and improve materials properties
- Defect control through growth condition variation and surface preparation

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Group IV molecular beam epitaxy

Our solid-source molecular beam epitaxial deposition capabilities are used in materials science research on strain layer heteroepitaxial growth of Group IV semiconductors. Collaborative work on strain relaxation mechanisms, surface segregation, interdiffusion, and strain-driven quantum dot self-assembly is envisioned. Growth on pre-patterned surfaces using CINT lithographic capabilities can be performed to examine directed self-assembly. Studies of stress and structural evolution in polycrystalline and amorphous films are also of interest. The capabilities that are available to CINT Users during jump-start operations include:

- UHV-MBE growth of Si, Ge, C, and their alloys;
- Video-RHEED acquisition
- Multi-beam optical stress sensor (MOSS)
- Maximum 2 inch wafer handling capability
- Growth and annealing up to 1000°C
- Atomic hydrogen supply

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Metallic multilayer synthesis

Our physical vapor deposition capabilities are used to synthesize nanoscale metallic multilayered materials where the individual layer thickness may be well controlled down to a nanometer. Depending on how many layers are deposited, the total thickness of the sample may vary from a micron to a couple tens of microns. Through appropriate masking techniques, the films could be pattern in shapes, e.g., as self-supported tensile samples. Collaborative work on stresses and mechanical behavior, fatigue, thermal stability, fracture, and creep of these multilayers as a function of nanostructuring dimensions is envisioned. Capabilities available include:

- Electron beam evaporation
- Magnetron sputtering

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Energetic neutral atom beam lithography

This facility offers low-temperature growth of thin film oxide and nitride materials and etching of polymeric materials with sub-100nm feature sizes. Energetic Neutral Atom Beam Lithography (ENABL) is particularly applicable to processing “soft” materials, organics, insulators, and semiconductors. A unique energetic neutral atom source produces a collimated beam of reactive atomic species (e.g. O and N) with high kinetic energies (1 to 5 eV) and is capable of delivering an extremely high flux ($\sim 10^{17}$ atoms/cm²sec) to a substrate over a large area (>10cm²). The facility includes atomic beam characterization, a full range of surface characterization techniques (XPS, AES, etc.), e-beam evaporators, and other thin film diagnostics. ENABL can be used to grow thin film oxide (Al₂O₃) and nitride (AlN, GaN, InN) materials having high crystallinity,

CINT Jump Start User Capabilities

epitaxy, and optical quality on a variety of substrates and for selectively etching of nanoscale features in polymeric films yielding sub-100 nm features with aspect ratios exceeding 25:1. Patterned oxide and nitride thin films can be directly grown in selective areas by combining etching with low-temperature deposition.

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Nano/Micro Fabrication

Electron beam lithography

A JEOL JBX-5FE Electron Beam Lithography System is resident in the Compound Semiconductor Research Laboratory (CSRL). This is a field-emission system operating at 50kV with a five inch stage. The minimum spot size is 5 nm and the minimum achievable feature size is order of 20 nm. Typical feature sizes are in the range of 50-250 nm. This tool, in association with other etch and deposition capabilities in the CSRL, provides powerful nanofabrication capabilities for a wide variety of materials and applications. Other relevant parameters include:

- Can handle nominally rectangular samples in the range of 5-25 mm;
- Can handle wafers of 2, 3, 4, and 6 inches in diameter
- Typical positive resist used is PMMA
- Typical negative resist is SAL-603 or NEB-31A

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Photolithography; Patterned metal and dielectric thin films; Plasma etch and ion milling

The micro system fabrication capabilities will provide researchers with distinctive platforms for investigating standard or hybrid materials. Our facility has an unrestricted tool set, which accommodates a wide range of substrates, films, and chemicals. We work closely with other centers/laboratories to allow integration of unique materials or processes into prototype micro/nano systems. The capabilities that are available to CINT Users during jump-start operations include:

Standard Micro fabrication

- Mask design
- Photolithography
- Metal deposition (E-beam evaporation and sputter deposition)
- Dielectric film growth and deposition (thermal oxidation, sputter deposition and plasma deposition)
- Plasma etching and ion milling
- Wet chemistries

CINT Jump Start User Capabilities

- Dicing saw
- SEM

Unique capabilities

- Fabrication of micro fluidic systems by configuration of channels above or below the substrate surface
- Packaging of micro fluidic systems (DRIE, anodic bonding and commercial dispensing system)
- Electroplating for thick film applications
- Micro stamping for benign materials

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Nanomaterials Synthesis

Polymeric monolayer systems

Surface properties are critical in many nanosystems, and the control of surface properties such as wetting, adhesion, and friction are of primary concern. Monolayer synthesis allows researcher to tailor surface properties utilizing small molecule organic synthesis and polymerization techniques. Either *in situ* or *ex situ* syntheses can be performed where appropriate and multilayers or gels may be produced using similar techniques. Capabilities that are available include:

- Monolayer design and formation on planar, particulate, chip-based, or other samples of inorganic oxides, non-oxidized metals, semiconductors, polymers, etc.
- Synthesis of functional coupling agents, in particular those with functionality.
- *In situ* modification of monolayer functionalities where desired functionalities lack compatibility.
- *In situ* growth of polymer monolayers and mixed polymer monolayers using free radical, ionic or coordination polymerization reactions.
- A suite of characterization methods to determine or verify monolayer functionality, structure, wetting properties, etc.

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Photocatalytic growth of nano-assemblies

Photocatalytic and catalytic methods are used to synthesize and characterize nanostructured materials composed of metals, semiconductors, and polymers. The advantage of catalytic reduction of metal ions over simple chemical reduction is that many metal atoms may be deposited at the site of the catalyst molecule. This allows considerable control over the metal deposition process by spatially organizing the catalyst molecules, and for a photocatalyst,

CINT Jump Start User Capabilities

controlling the subsequent rate of reduction by the controlling light intensity. The resulting structures typically are composite nanoassemblies composed of the templating media (self-assembled surfactant, polymer, silicates, etc.), the active photocatalyst, and the grown metal, semiconductor, or polymer. To date, we have focused mostly on metal nanostructures, but future interests include controlled synthesis of semiconductor and polymer nanostructures

The capabilities available to CINT Users include:

- synthetic chemical processes
- electron microscopy
- dynamic light scattering

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Mesoporous materials

Mesoporous solids are materials with a precise periodic structure of porosity at the nanoscale. Our technique for synthesizing these materials is based on evaporation-induced self-assembly, in which evaporation during dip- or spin-coating enriches a depositing film in silica and surfactant, thereby inducing the continuous self-assembly of silica/surfactant mesophases. The evaporation conditions and, hence, the structure of the films can be precisely controlled for applications such as gas separation membranes, low- k dielectrics, and chemical concentrators for sensors. This self-assembly process can also be used to mimic the hard/soft laminated construction of natural materials like sea shells (*nacre*). Micelles are also used to organize both organic precursors and hydrophilic inorganic precursors into liquid crystalline mesophases. This simple evaporation induced route is a powerful and versatile means of nanomaterials and nanocomposite assembly. We want to obtain an improved understanding of such assembly processes to predict and tailor the molecular-level construction of hierarchical structures with unique physical, optical, electronic and chemical properties. In addition, we are interested in strategies to extend this basic synthetic paradigm to examine the development of optically and electronically active, hybrid nanostructures, to investigate new approaches for the realization of superhydrophobic surfaces, and to utilize bio-compatible chemistries to fabricate synthetic supports for living bilipid membranes and whole cells. The capabilities that are available to CINT Users during jump-start operations include:

- Lab for synthesizing mesoporous films by dip-coating, spin-coating and aerosol spray;
- Membrane characterization
- Ellipsometry
- Characterization of porosity

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Colloidal assembly/Surface templating:

Our facilities are aimed at understanding interparticle forces, developing the science of colloidal crystal (CC) assembly, and developing tunable behavior in CCs through use of active materials and templating. Colloidal crystals, i.e. space-filling assemblies of colloidal particles with long-range translational symmetry, can exhibit photonic band gap (PBG) behavior normally dictated by refractive index contrast, symmetry, and lattice constant. The introduction of suitable photo-, electro- or chemically active organic or hybrid material phases into the interstices or particles of a colloidal crystal, however, can enable the reversible modulation of the structural and optical characteristics of the CC through appropriate stimulation. Our laboratory is involved with synthesis, templating, and assembly of a variety of organic and inorganic particles. Characterization methods include measurements of particle size distribution, interparticle forces, rheology, contact angle, aggregation behavior, and optical properties. Microcontact printing (μ CP) of a variety of self-assembled monolayers has been used to direct or template formation of ordered assemblies. In addition, electroactive particles and photoactive interparticle linking agents have been developed and characterized. Capabilities available to CINT Users during jump-start operations include:

- Wet laboratory facilities for particle synthesis by methods including Stöber sphere synthesis and rapid liquid phase nucleation
- DT1200 acoustic spectrometer for particle size distribution and Zeta potential measurement
- Haaka RS300 rheometer for rheological characterization
- Hach turbidimeter for aggregation rate characterization
- Glove box chemical synthesis, dip coating, and spin coating
- Electrical and optical property characterization suite
- Microcontact printing of SAMs via PDMS elastomeric stamps

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Oxide nanomaterials

The focus of our work is developing and exploiting our understanding of materials chemistry to control the structure and properties of the materials we synthesize. Our work is concentrated on, but not limited to, developing novel metal alkoxides with the hydrolysis and condensation rates controlled by the types of ligands used. A majority of the compounds that have developed are unique (> 400 compounds) and are not commercially available, which allows us to enter a realm of materials synthesis that has not been previously addressed. Once fully characterized, these compounds are used for fabricating thin films by either solution routes or metallo-organic chemical vapor deposition (MOCVD). Powders can also be generated from these solutions by removal of the volatile components or through precipitation mechanisms. In depth understanding and characterization of the various systems investigated permits the use of designer molecules to reduce synthesis time, improve solution stability, lower processing temperatures, and reduce heat-treatment times. This approach can also facilitate uniform incorporation of dopants, improve microstructures and material properties, and generate new or simpler routes to fabricating materials. We are now investigating nanoparticle synthesis using

CINT Jump Start User Capabilities

these metal alkoxide and mixed-metal alkoxide precursors. The capabilities that are available to CINT Users during jump-start operations include:

- Facilities for synthesizing metal alkoxides, including air sensitive materials, and nanoparticles.
- FTIR, UV-Vis spectrometers for characterizing materials.
- Ability to crystallize and do structural characterization and refinement of precursors.

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Colloidal synthesis: Semiconductor, metal, and magnetic nanocrystals

In our laboratory we emphasize the preparation of “high-quality” semiconductor, metal, and magnetic nanocrystals. We define success in part by our ability to control size dispersity, particle crystallinity, particle stability, and particle optical/electronic/magnetic properties. These criteria comprise our definition of “high-quality.” Typically, our nanocrystals are prepared with a target functionality in mind. We work closely with physicists and spectroscopists who, through their advanced characterization tools, inform our synthetic work. We strive to understand, for example, the effects of particle size, particle shape, and particle surface structure and functionalization on nanocrystal properties and, subsequently, to optimize these properties. We focus on the preparation of new compositions (core and core/shell materials; UV to visible to infrared absorbers/emitters; ferromagnetic to superparamagnetic nanoparticles, etc.), new shapes (isotropic to highly anisotropic), composite materials (*e.g.*, high-density nanocrystal/sol-gel processible blends), and biocompatible nanocrystals (water-soluble and functionalized for binding to various biomolecules), as well as self- and directed-assembly of films and composite structures. Nanocrystal chemical-precursor development and ligand/surfactant development are pursued when necessary.

Capabilities available include:

- Facilities for synthesizing and assembling colloidal nanocrystals, and facilities for thin-film preparation, including air-sensitive handling methods, LB trough, and multi-gun sputtering system
- Expertise in inorganic, organic, and materials chemistry
- In-lab (and partner-lab) facilities for microstructural, optical and magnetic-properties characterization of nanoscale systems, including UV-Vis, near-IR FTIR, fluorimeter, AFM, NSOM, MFM, TEM, optical microscopy, and ultrafast laser spectroscopy

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Chemical synthesis of nanoscale electronic materials

Inorganic and organic synthetic chemical procedures leading to nanoscale electronic and optically active building blocks, including semiconductor quantum dots, metal nanoparticles and conjugated organic polymers are available. Capabilities include:

CINT Jump Start User Capabilities

- Colloidal nanocrystal synthesis, as well as chemical-precursor and ligand development (see above)
- Synthesis of water soluble conjugated polymers
- Preparation of nanostructured fibers and composites, including chiral composites, involving conjugated polymers
- Synthesis and characterization of metal nanoparticles
- Stabilization of metal nanoparticles in solution

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Thin-film preparation

Preparation and characterization of thin-films using chemical assembly routes is possible for many different types of materials and substrates. The major focus of activities is on different self-assembly routes to thin-film materials, and on preparative strategies that involve combinations of processing (spin or dip-coating; post-deposition patterning) with self-assembly strategies. Specific capabilities available include:

- Dip-coating and spin coating of soluble polymers, including multi-layer assemblies of water soluble conjugated polymers and other poly-electrolytes;
- Langmuir-Blodgett capabilities, including multi-layer deposition and Brewster-angle microscopy characterization;
- Surface cleaning tools and techniques;
- Incorporation of nonlinear optical chromophores into amphiphilic assemblies and Langmuir-Blodgett films;
- Self-assembled monolayer formation on metal and oxide surfaces;
- Thin-films of mesoporous and mesostructured silica;
- Patterned films of electronically active nanoscale building blocks (conjugated polymers, fullerenes);
- Spatial patterning of organic thin films using masked deep-uv exposure

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Intermetallic and oxide synthesis and crystal growth

Diverse capabilities and resources exist for the synthesis, often in single-crystal form, of a variety of intermetallic and oxide compounds and alloys. Our particular interest is in those materials that display emergent nanoscale phenomena and novel ground-state properties. These synthetic capabilities are also available as resources to provide source materials for related CINT activities.

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Organic synthesis and crystal growth

The long-standing promise of organic materials as the basis for a new class of large-area and flexible electronic devices is starting to be realized - already thin film transistors exhibit performance on par with amorphous silicon. However, little is known about fundamental transport properties, in particular the origin and controllability of deep charge-trapping states. Future studies will require single crystals of exceedingly high purity, which are only achievable through Bridgeman zone-refining techniques. Capabilities include:

- Bridgeman zone refining furnace
- High voltage semiconducting characterization
- Transport characterization to helium temperatures

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Polyoxoniobate chemistry

Polyoxometalate (POM) chemistry, dominated by metal-oxo clusters of tungsten, molybdenum and vanadium has relevance to a diversity of technologies including protein binding, detection and precipitation, anti-viral applications, catalysis, anti-corrosion, and a variety of electrochromic and -optic applications. We have recently developed a general synthetic procedure for the synthesis of polyoxoniobates (Nyman *et al.*, *Science* 2002), which opens up a whole new realm of possibilities for chemical and structural diversity and applications of POM materials. Polyoxoniobate clusters differ from their tungstate, molybdate and vanadate cousins in that they are base stable and have a much higher charge to surface ratio. We are using spectroscopic techniques such as multinuclear NMR to understand how the niobates differ from the other polyoxometalates in stability and reactivity, and how these differences can be exploited in new applications. We are also interested in using molecular modeling to understand how composition of polyoxoniobates may influence geometry of clusters. Some applications we are investigating for polyoxoniobates include nuclear waste processing, development of ordered arrays with collective properties, and pathogen sequestration and binding.

The capabilities that are available to CINT Users during jump-start operations include:

- Synthetic lab including apparatus for air-sensitive manipulations
- Autoclave reactors
- Characterization tools include X-ray diffractometer, ICP-MS, Scanning Electron Microscope with Energy Dispersive Spectroscopy, Ion Chromatography, Scanning Tunneling Microscopy and Atomic Force Microscopy. Also available is surface area analysis, thermogravimetric and differential thermal analysis, and infrared spectroscopy.

CINT Jump Start User Capabilities

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Biomaterial Synthesis

Lipid membranes and self-assembled films

An assortment of capabilities exists for the synthetic preparation of functionalized amphiphiles and their incorporation in lipid vesicles, supported lipid membranes, self-assembled monolayers on silicon surfaces, and Langmuir films. The self-assembled films can be interrogated with a variety of spectroscopic techniques, which include dynamic light scattering, fluorescence microscopy and spectroscopy, NMR, and XPS, and at the nanoscale via *in situ* AFM imaging and TEM. Interactions of metal ions, small molecules, proteins, and whole cells against functionalized films have been previously explored. The capabilities that are available to CINT Users during jump-start operations include:

- Wet laboratory facilities for the synthesis and characterization of functionalized amphiphilic molecules
- Liposome preparation via sonicators and extruders
- Langmuir troughs for monolayer and multilayer film preparation
- Inverted fluorescence microscope coupled with intensified CCD camera and CCD spectrometer for simultaneous imaging and spectroscopic characterization of Langmuir monolayers
- Temperature controlled *in situ* AFM for nanoscale imaging under varying environmental conditions
- Microcalorimetry to measure binding energies of protein association at lipid membrane surfaces
- Fluorescence recovery after photobleaching (FRAP) characterization of lateral mobility in substrate-supported lipid membrane assemblies
- Brewster angle microscopy for characterization of thin films
- Generation of patterned hybrid and supported bilayer assemblies on derivatized substrates

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Bio-inspired and Bio-compatible Materials

The biomaterials synthesis capabilities will enable researchers to isolate, engineer, and integrate biological molecules with nanoscale synthetic materials and systems. Because native biological molecules are, in general, poorly suited for integration with synthetic systems, we focus upon engineering biomaterials specifically designed to function in synthetic nanosystems. Additionally, functionalization of biological molecules will be studied with respect to developing strategies for integrating living and non-living components that have a common interface. The

CINT Jump Start User Capabilities

capabilities that are available to CINT Users during jump-start operations include:

- Isolation of genomic DNA, RNA, and plasmids from a variety of sources such as bacteria, viruses, and eukaryotic cells
- Growth and maintenance of a range of organisms (e.g., thermophiles, halophiles, etc.)
- Recombinant DNA cloning and expression in prokaryotic and eukaryotic systems
- Genetic engineering using reverse transcription, the polymerase chain reaction (PCR) and site-directed mutagenesis (SDM)
- Expression, purification, characterization, and functionalization of native and recombinant proteins
- Synthesis and functionalization of bio-compatible nanocrystal optical and magnetic tags (semiconductor and metal nanocrystals)

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Biochemical techniques and instrumentation

Several biochemical approaches and instrumentation are available that can be applied towards characterization of biomaterials and/or related compounds. These range from protein separation strategies to mass spectrometry based characterization. The available capabilities include:

- Capillary electrophoresis (autosampling capable)
 - UV/Vis Detection
 - Optical waveguide based two color LIF detection
 - ESI-MS interface for mass analysis of analytes
- ESI-MS and MALDI-TOF MS
- Gel electrophoresis (SDS-PAGE, IEF, 2DE, agarose etc.)
- Microchip based multi-dimensional solution electrophoresis with LIF detection
- Image analysis, spot excision, automated proteomics tools
- PCR, Capillary sequencers
- Standard biochemical laboratory equipment

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Ligand development

Selection of high affinity binding ligands from large diverse phage display libraries can be performed. Libraries are either antibody based (single chain fragments in particular) or fluorobodies. The latter are experimental binding ligands still under development which combine the advantages of antibodies (specific high affinity binding) with green fluorescent protein (intrinsic fluorescence, high solubility, expression and stability). In collaboration and upon supply of appropriate targets we can select binding ligands. Presently we are attempting to see if such ligands can be selected against materials of interest in nanotechnology. Potential technologies of interest to CINT users:

CINT Jump Start User Capabilities

- Phage-display methods
- Selection of specific scFvs or fluorobodies

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Scanning Probe Microscopy

Atomic force microscopy, near field and fluorescence imaging

Full imaging and characterization of biological interfaces such as membranes and model lipid systems will be available. The facility will include atomic force microscopy in ambient and fluid environments, in tandem with far-field fluorescence imaging and apertureless near-field imaging.

Facilities that will be available include:

- Atomic force microscopy
- Atomic force combined with fluorescence microscopy, including apertureless near-field techniques
- Fluorescence microscopy

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Atom tracking scanning probe microscopy

Our atom-tracking scanning tunneling microscopes (STM) are used to study the motion of individual atoms, molecules, or clusters over crystal surfaces as a function of temperature. In atom-tracking mode the STM probe tip is “locked” onto a diffusing adsorbate using lateral, X-Y, feedback and the diffusion path is continually monitored. This mode increases the time resolution of kinetic measurements by a factor of 1000 over conventional STM imaging.

Facilities that will be available include:

- Atom-tracking STM studies (selected metals, Si, Ge)
- Kinetic modeling of surface diffusion

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Interfacial force microscopy

The Interfacial Force Microscope (IFM) is a unique, Sandia-developed scanning force-probe technique featuring a mechanically stable, self-balancing force sensor, which has the capability to quantitatively measure normal and lateral interfacial forces at the nanoscale. Current interests for IFM applications include: (1) studies of the fundamental mechanisms underlying molecular-

CINT Jump Start User Capabilities

level friction for functionalized surfaces; (2) studies of the nanomechanical properties of self-assembled films, interphase materials and solid surfaces, including molecular films, polymers and metals; and (3) studies of the fundamental aspects of charge transfer in various systems under including non-contact, contact and under applied stress. The capabilities that are currently available include:

- IFM instrumentation with broad facilities for tip and sample preparation and analysis, molecular self assembly and environmental control;
- Controlled Imaging capabilities for quantitative analysis of, e.g., morphology, complex-modulus mapping, conductance, friction, etc.
- Control of tip material, size, shape and chemical functionality;
- Considerable experience in data analysis to obtain fundamental materials properties.

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Optical Microscopy/Spectroscopy

Optical spectroscopy

Optical spectroscopy is an essential tool for the characterization of many nanomaterials. Although these techniques are not capable of directly resolving individual nanostructures they can be used for investigating important nanoscale processes such as energy transfer and plasmonic transport. The available capabilities include:

- Optical spectroscopy, UV-Vis, Fluorescence Spectroscopy, FTIR
- Raman and Infrared Spectroscopy
- Ellipsometry
- Attenuated total reflection (ATR) spectroscopy;
- Thin-film waveguide characterization;
- Light scattering
- Interferometry

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Optical microscopy

Advanced spectroscopic techniques can be combined with optical microscopy to provide a suite of tools for characterizing the spatially dependent properties of nanoscale materials. The available capabilities include:

- Microscopy: light, fluorescence, and high-resolution hyper-spectral
- Single-molecule detection techniques
- Near-field scanning optical microscopy, combined with both cw and transient absorption spectroscopy

CINT Jump Start User Capabilities

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Ultrafast laser spectroscopy

Ultrafast spectroscopy provides important information about the excitation and relaxation dynamics occurring in nanomaterials. A suite of ultrafast excitation and diagnostic capabilities, spanning wavelengths from the ultraviolet to the far-infrared are available for dynamic nanoscale characterization. These capabilities enable coherent quantum control experiments, as well as experiments for dynamic materials characterization. The available capabilities include:

- Femtosecond broadband transient absorption spectroscopy (infrared to ultraviolet)
- Time-resolved femtosecond photoluminescence
- Degenerate and non-degenerate four-wave mixing
- Optical pump/terahertz probe spectroscopy
- Ultrafast scanning tunneling microscopy
- Ultrafast optical characterization at photonic wavelengths (1.55 μm)
- Femtosecond pulse shaping capability with 20 fs optical pulses and electric field measurement diagnostics
- Cryogenic and magnetic field in combination with ultrafast spectroscopy

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Electron Microscopy

Low energy electron microscopy

The Low Energy Electron Microscope (LEEM) is a unique and versatile surface microscope that can be used to view dynamic processes on surfaces in real time with a spatial resolution of 7-8 nm and a depth resolution of one atomic layer. The LEEM provides high image contrast between regions on a surface with different atomic structures or chemical compositions. Because it is a non-scanning microscope, dynamic processes can be observed with a time resolution limited only by the video recording rate of the image acquisition system. Current interests for LEEM applications include: 1) studies of the fundamental mechanisms underlying self-assembly and pattern formation on solid surfaces, 2) studies of the evolution of surface morphology including thermal smoothing mechanisms, and 3) studies of the fundamental aspects surface phase transitions and surface chemical reactions.

Specific capabilities of the LEEM include:

- Ultra-high vacuum ($<1 \times 10^{-10}$ Torr) in main chamber
- Sample cleaning by ion bombardment
- Surface characterization by Auger Electron Spectroscopy

CINT Jump Start User Capabilities

- Images can be recorded with the sample at temperatures from 200 K to 1800 K
- Images can be recorded with a flux of atoms or molecules impinging on the surface

For more information, contact:

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Nuclear Magnetic Resonance

Nuclear magnetic resonance of ceramics

NMR instrumentation is available for investigations of local chemistry, physics, structure and domain size in nanomaterials. We are interested in interface chemistry, self assembly processes and structural studies of novel materials. Both high-resolution solution and solid-state NMR characterization methods are possible. The capabilities that are available to CINT Users during jump-start operations include:

- High field Bruker Avance 3-frequency channel 600 MHz NMR spectrometer with both solid state and solution capabilities including: multinuclear MAS probes, high speed (~ 35 kHz) ^1H -X MAS probe, wide line state probes for low frequency nuclei (^{67}Zn , $^{49,47}\text{Ti}$, ^{89}Y , ^{93}Nb), high resolution multi-nuclear solution probes with pulsed-field gradients and dedicated ^{19}F - ^1H high resolution NMR probes
- Bruker Avance 3-frequency channel 400 MHz NMR spectrometer dedicated to solid-state NMR investigations. Capabilities include: multinuclear MAS probes, triple resonance MAS probe, high temperature (~ 650 °C) MAS probe, high speed (~ 35 kHz) ^{19}F -X MAS probe, MQMAS, DOR and static wide line NMR probes
- Bruker DRX400, 3-frequency channel high-resolution spectrometer. Capabilities include: multi-nuclear high-resolution and triple resonance probes with triple axis pulse field gradients, a dedicated ^{29}Si probe, a dedicated $^{203,205}\text{Th}$ probe, diffusion measurements and standard VT capabilities

For more information, contact:

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Mechanical Properties

***In-situ* mechanical testing**

The *in-situ* mechanical testing capability is focused on studying the fundamental correlations between microstructural evolution and both applied and intrinsic stresses in thin film. A primary objective of this work is to determine the stress relaxation mechanisms that dominate in the submicron to nano-meter regime. This includes the study of the effect of both decreased external dimensions and grain size on stress relaxation processes, e.g. dislocations, fracture, grain-boundary sliding. A second objective is to determine the mechanisms inducing stress during

CINT Jump Start User Capabilities

electrodeposition of metals. Of particular interest is the link between the nucleation morphology and the final film stress. The following capabilities will be available:

- *In-situ* stress and polarization measurements during electrodeposition of Ni, Ni alloys, and Cu and their alloys
- Development of an *in-situ* MEMS tensile tester for quantitative stress analysis of free-standing metal films during TEM imaging (with a concentration on examining the effects of scale on deformation processes)
- Electro-deposition and physical phase deposition of metals, contact mask lithography, and e-beam lithography

For more information, contact:

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Mechanical properties from near-surface layers

The mechanical properties of thin films and layered materials or very small volumes of materials cannot be measured using conventional techniques. Nanoindentation methods have been developed to probe materials at depths of tens of nanometers over regions with dimensions of hundreds of nanometers. Using continuous stiffness measurement, we have the capability to measure changes in mechanical properties as a function of depth. Substrate or layering effects can also be examined. We have also measured material length scales and size effects resulting from dislocation concentrations and dislocation interactions with other structural defects. Changes in material properties resulting from impurities, second phase inclusions or engineered nanostructures can also be measured.

Specific capabilities include:

- Nanoindenter with continuous stiffness measurement

For more information, contact:

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Transport

Physical characterization at extremes of parameter space

This characterization capability includes the measurement of transport properties and magnetic susceptibility as functions of temperature, pressure and magnetic field. Electrical resistivity and potentially Hall effect can be measured on solid samples at pressures to 1.6 GPa, temperatures from 0.35 to 300 K and in magnetic fields to 8 T can provide information about electronic scattering mechanisms and carrier density. Magnetic susceptibility/magnetization measurements give magnetic properties of nano-sized particles and are performed in the temperature range $2 < T < 350$ K, in fields to 7 T and at pressures to 0.6 GPa. Specific heat on mg-size solids samples can be measured at atmospheric pressure in fields to 9 T and in the temperature range 0.35 to 300

CINT Jump Start User Capabilities

K. This measurement gives the electronic density of states, entropy as a function of temperature, and characteristic phonon frequency.

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Low temperature electronic transport

The electronic properties of nanoelectronic structures exhibit quantum mechanical and interaction effects at low temperatures and high magnetic fields. Devices designed to explore these effects can be fabricated on compound semiconductor heterostructures using standard microfabrication techniques. The primary capability for rapid transport characterization of the fabricated devices and longer-term studies of nanoelectronic devices is a top-loading cryostat with T=0.3 Kelvin base temperature and 16 Tesla magnetic field configured for DC transport measurements.

For more information, contact:

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Theory and Simulation

Advance of computing technologies and modeling approaches have enabled detailed studies of collective and cooperative materials phenomena at various length scales. However, it is well recognized that fundamental understanding of the behaviors of nanostructured materials will not be addressed by simple extensions of current theoretical methods that are focused on either atomic or macro scales, but will require bridging the gap between these scales with new concepts, new modeling frameworks and new simulation schemes. The Theory and Simulation thrust area will support CINT Users by providing expertise in a number of fields including theory, predictive capability development, multiscale material modeling and large scale computing. The capabilities that are available to CINT Users during jump start operations include:

- Simulation tools for computational materials science
 - LAMMPS: a parallel molecular dynamics code for classical atomistic and coarse grained level simulations
 - PARADYN: a parallel molecular dynamics code for embedded atom models of metals
 - TRAMONTO: a parallel, classical density functional theory code for atomic and polymeric fluids
 - A variety of tools for visualization and analysis
- Modeling and simulation of soft nanomaterials and biomolecules
- Modeling and simulation of hard nanomaterials
- Modeling of interfacial phenomena, self assembly, and granular materials

CINT Jump Start User Capabilities

- Theory and modeling of scanned and imaging nanoscale probes

For more information, contact:

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Leveraged Facilities

Ion Beam Materials Laboratory

The core of the laboratory consists of a 3.2 MV Pelletron[®] tandem ion accelerator, a 200 kV ion implanter, and an 8 kV plasma immersion ion processing system. The tandem accelerator has five beam lines with a series of experimental stations that support various research programs. The operation of IBML and its interactions with users are organized around core facilities and experimental stations. The IBML provides and operates the core facilities, while supporting the design and implementation of specific apparatus needed for experiments requested by users of the facility. This results in a facility with competencies in routine ion beam experiments and the versatility to cater to the individual researchers needs. Detailed information is available at <http://www.lanl.gov/mst/ibml/>. Available capabilities include:

- Ion implantation with various ion species
- Plasma immersion ion surface treatment of irregular shaped large samples
- High energy particle irradiation of various materials (Gases, Liquids, and Solids)
- Routine Ion Beam Analysis Techniques:
 - Rutherford backscattering spectrometry
 - High energy elastic scattering spectrometry
 - Elastic recoil detection analysis or Forward recoil spectrometry
 - Nuclear reaction analysis
 - Particle induced X-ray emission spectroscopy
 - Ion channeling with a 5-axis goniometer
- Nuclear microprobe for ion beam microanalysis and microfabrication
- Joint target chamber for Implanter and Accelerator to conduct in situ ion beam modification and analysis

Users may request the use of this capability for sample measurements alone or as part of a scientific effort involving staff collaboration and, if appropriate, other CINT capabilities. For facility information in general, please contact Dr. Y.Q. Wang. For scientific collaborations involving this capability, please contact either Dr. Wang or Dr. Nastasi.

For more information, contact:

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CINT Jump Start User Capabilities

MicroElectroMechanical Systems

The SAMPLES™ Program is a way to provide customers access to the revolutionary MicroElectroMechanical System (MEMS) technologies developed at Sandia National Laboratories. The objective of the SAMPLES™ Program (Sandia Agile MEMS Prototyping, Layout Tools, Education and Services) is to enable customers to develop their own innovative products by leveraging advanced design, fabrication, and characterization technologies originally developed for National Laboratory applications. Participants can attend short courses, design new devices, and have those designs fabricated in our state-of-the-art fabrication facility.

For more information, contact:

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Nuclear Magnetic Resonance Facility

The NMR facility in the Bioscience Division at LANL includes two Bruker Avance 500 MHz spectrometers and a Bruker AMX-400 MHz spectrometer. All three are high resolution NMR's capable of observing NMR signals from liquid samples. The Avance 500 MHz spectrometers are equipped with pulsed field gradients and have four channels and inverse triple resonance probes for simultaneous pulsing on ¹H, ¹³C, ¹⁵N and ²H. In addition, we have seven probes that allow observation of all NMR active nuclei. The AMX-400 has high power gradients and is capable of micro imaging.

For more information, contact:

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Electron Microscopy Laboratory

The EML facility contains two scanning electron microscopes (SEM), two analytical transmission electron microscopes (TEM), a high-resolution transmission electron microscope (HREM), specimen preparation equipment, optical microscopes, a digital imaging and analysis work area and a photographic darkroom.

Detailed information is available at <http://www.lanl.gov/mst/emf/>. Specific capabilities include:

- Philips XL30F Orientation Imaging Microscopy System (SEM/OIM)
- JEOL 6300 FX Field-Emission Gun SEM
- FEI Tecnai F30 Analytical TEM/STEM
- JEOL 3000F Field-Emission Gun High-Resolution TEM
- Philips CM-30 Analytical Electron Microscope
- Specimen Preparation Facility

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April 14, 2004

CINT Jump Start User Capabilities

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Scanning Probe Microscopy Facility

Detailed information is available at <http://www.lanl.gov/mst/SPML/>. Specific capabilities include:

- Variable Temperature Ultrahigh Vacuum Scanning Probe Microscope System (40K-1400K) with AFM, in-situ epitaxial growth, RHEED, Auger and LEED
- Ultrahigh Vacuum Scanning Tunneling Microscope System with Ballistic Electron Emission Microscopy, LEED and Auger Electron Spectroscopy
- Digital Instruments Nanoscope IIIA Scanning Probe Microscopes with AFM, MFM with external fields, Electric Force Microscopy, Kelvin Probe Force Microscopy, Tunneling Atomic Force Microscopy, in-situ tensile stressing
- Multimode Scanning Probe Microscope with Atomic Force Microscopy, heating capability ($\geq 250\text{ C}^\circ$), Magnetic Force Microscopy, Electric Force Microscopy, Piezo Response Force Microscopy, Scanning Tunneling Microscopy, contact mode AFM in liquids
- Room and Low Temperature (77K or 4K) MR head Magnetic Imaging

For more information, contact:

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National User Facilities

Los Alamos Neutron Scattering Center – LANSCE

For detailed information see: http://lansce.lanl.gov/index_ext.htm

National High Magnetic Field Laboratory – NHMFL

For detailed information see: <http://www.lanl.gov/mst/nhmfl/>

Combustion Research Facility – CRF

For detailed information see: <http://www.ca.sandia.gov/CRF/>