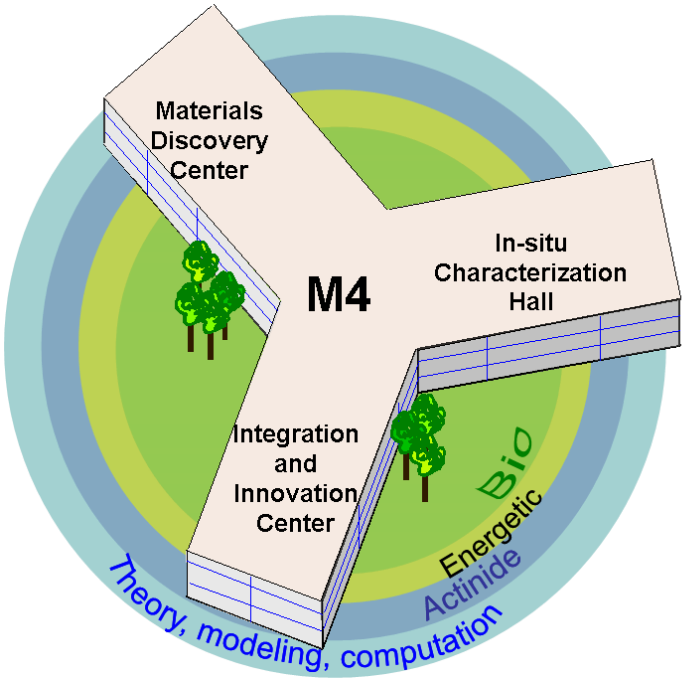
Materials Discovery: The MaRIE-M4 Facility



T Mark McCleskey (focus lead)
David Teter (Chairman of the Board)

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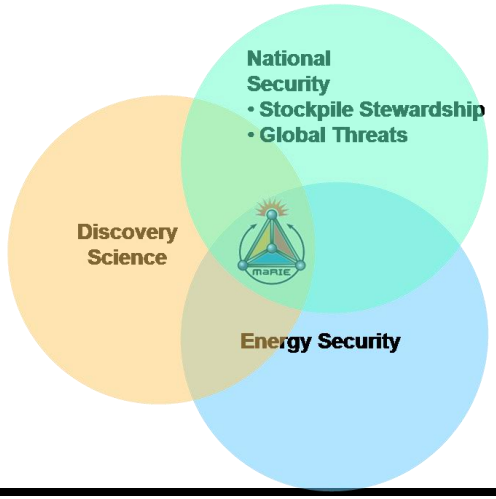
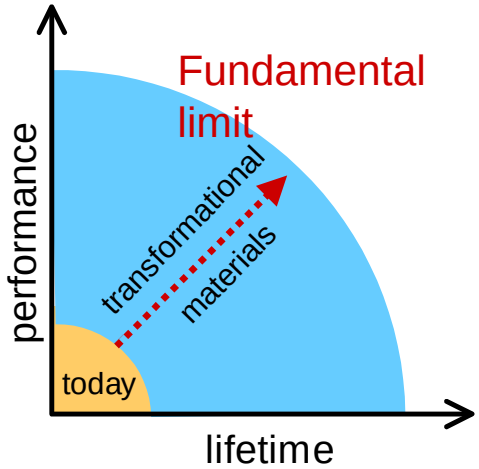
MaRIE: Materials R&D Capability for 21st Century National Security and Energy Security



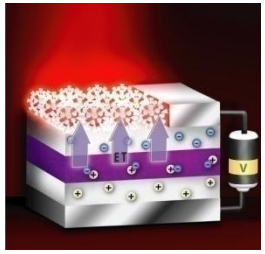
MaRIE addresses materials challenges across missions

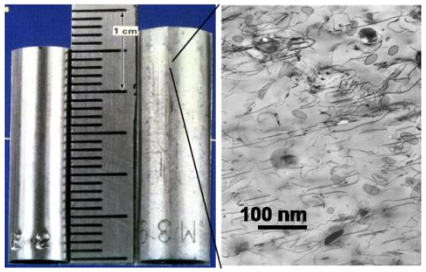
MaRIE enables the transition from "observation" to "control"



MaRIE will transform science of microstructure, interfaces, and defects

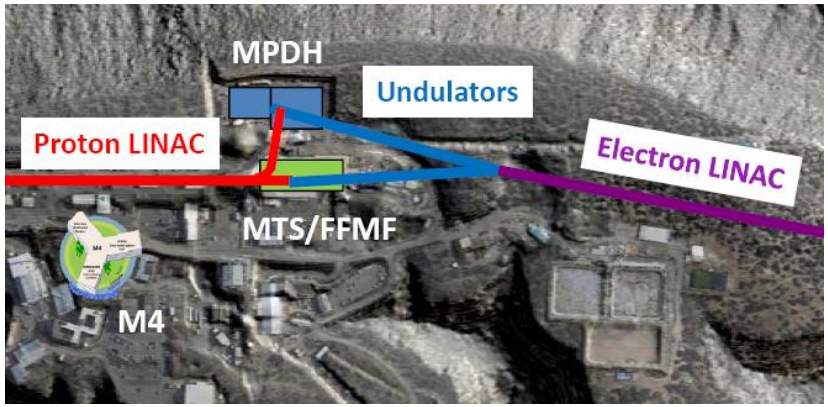
Next-generation solar cell architecture





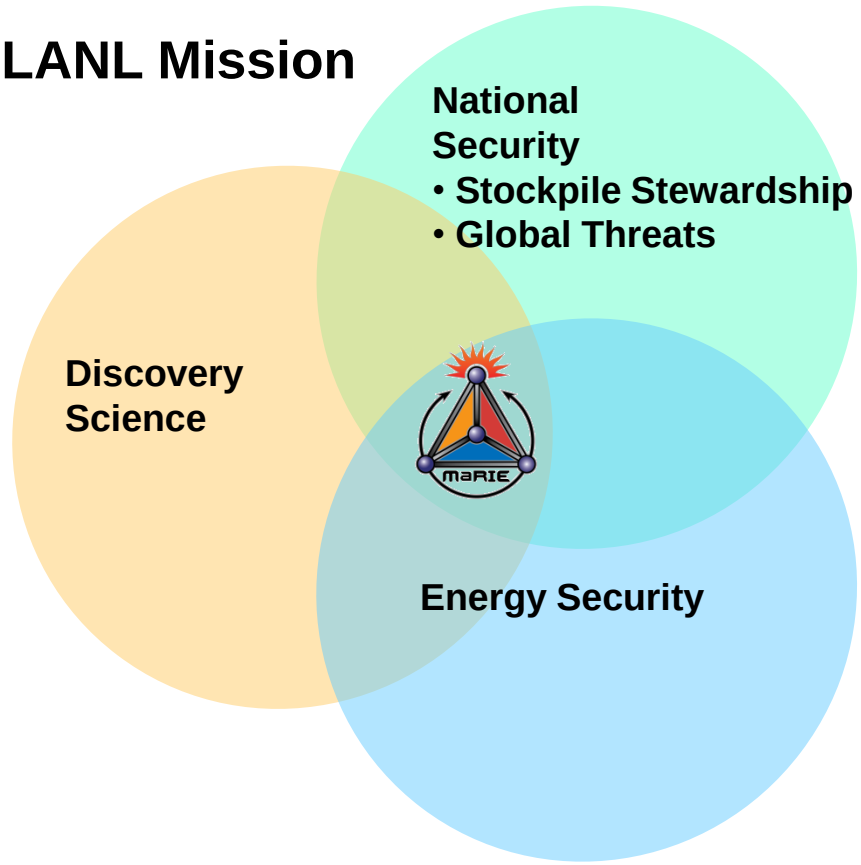
Radiation-induced swelling

MaRIE provides tools for transformational materials performance in extremes



MaRIE decadal research frontiers and challenges to address Los Alamos' national security missions

LANL Mission



National Grand Challenges

- **Transform the nuclear weapons enterprise: Define process-aware materials performance**
- **Close the 10 TW Gap between the energy we have and the energy we need: From fission & solar to fusion**

Materials Matter!
Material Requirements Central to National Grand Challenges

Materials Recognized as a Core LANL Capability

Enabling Materials-Centric National Security Science for the 21st Century

MaRIE - transformational advances in materials performance in extremes

First x-ray scattering capability at high energy and high repetition frequency with simultaneous charged particle dynamic imaging

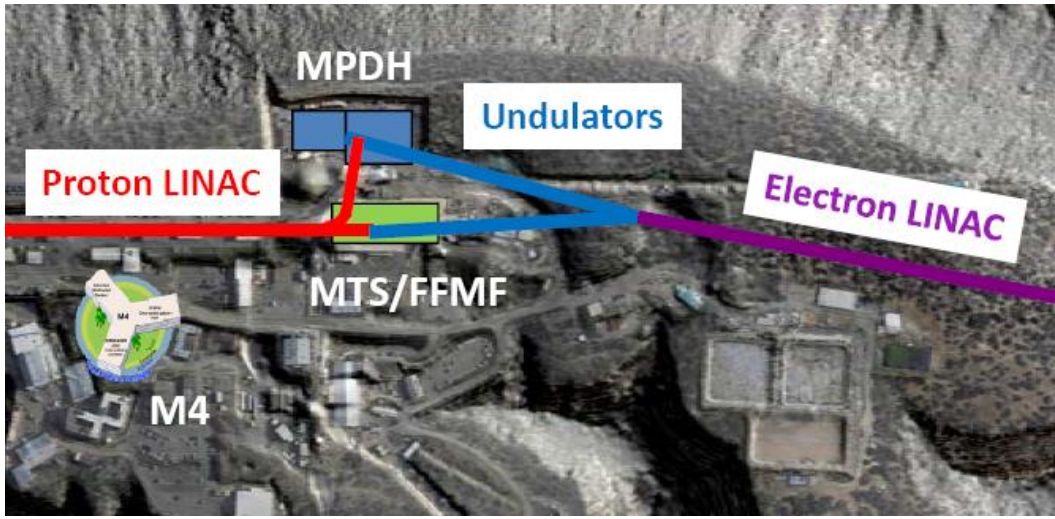
(Multi-Probe Diagnostic Hall)

Unique in-situ diagnostics and irradiation environments beyond best planned facilities

(Fission and Fusion Facility)

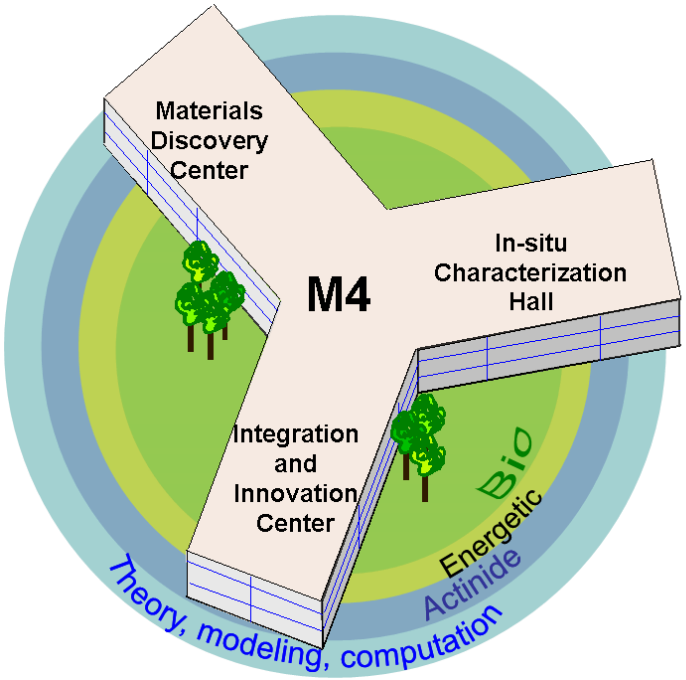
Comprehensive, integrated resource for materials synthesis and control, with national security infrastructure

(M4 Facility)



MaRIE will provide unprecedented international user resources

Materials Discovery: The MaRIE-M4 Facility



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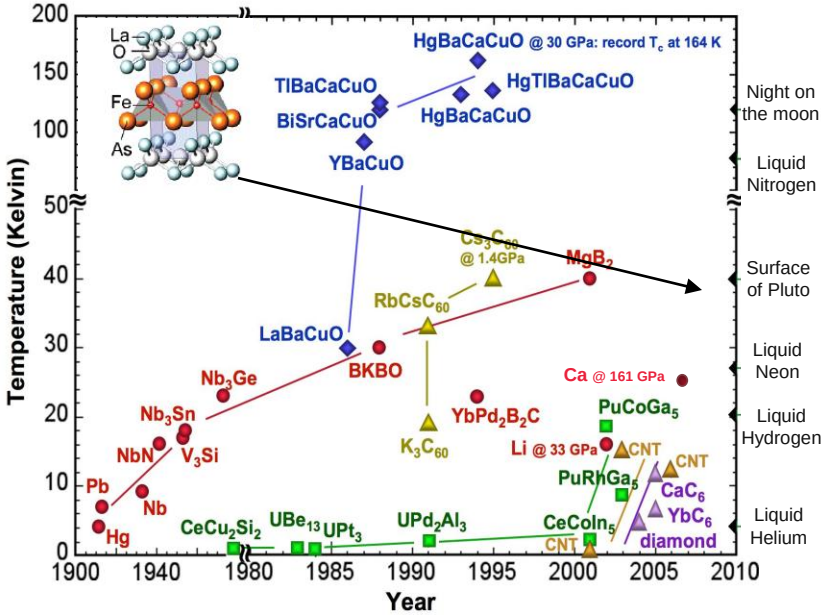
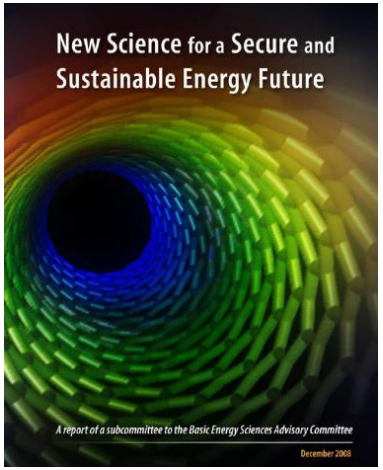
Observation to Control Science

- **Accelerated materials discovery - key to future technologies**
- **Process aware materials performance**
- **Composites materials of the future require improved theory**
  - Interfaces, defects and boundaries
- **Theory verification will occur by controlled synthesis**
  - Crystalline materials needed initially



Challenge of Materials Discovery Requires Integration



- **The community has identified the challenge of control to drive discovery**
  - ***Control*** material processes at the level of electrons.
  - ***Design*** and perfect atom- and energy-efficient synthesis of revolutionary ***new forms of matter*** with tailored properties.
  - ***Control*** properties of matter that emerge from complex correlations of the atomic or electronic constituents.
  - ***Master*** energy and information on the nanoscale to ***create new technologies*** with capabilities rivaling those of living things.
  - Characterize and ***control*** matter away— ***especially very far away —from equilibrium****?*

- **Recognized need for an integrated approach**
  - National Academy Study (2008): Assessment of and Outlook for New Materials
  - BES's Energy Frontier Research Centers
- **Integration across disciplines and length scales**
  - Produce *materials with precisely controlled defects*
  - Develop *multi-component, multi-functional materials*
  - Develop entirely new classes of materials and *innovative material architectures*

Customer Basis

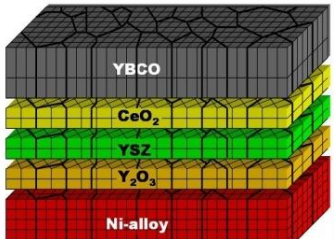
- **BES**
  - New science for a secure and sustainable energy future
    - Advanced nuclear energy systems
    - Materials under extreme environments
    - Superconductivity
    - Electrical energy storage
    - Solar energy utilization

    Control science for accelerated materials discovery
- **Defense Programs**
  - Process aware materials performance
- **OFES/ONE**
  - Materials for advanced fission/fusion reactors by design
- **Threat reduction** (DNDO, DARPA, DHS, INTEL …)
  - New materials for radiation detection
  - Integrated material systems

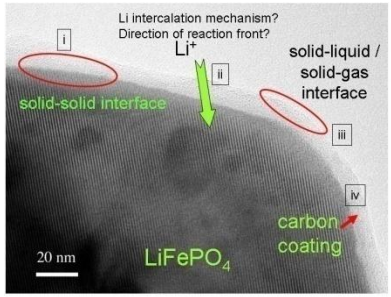
Accelerating Materials Discovery &
The Transition from Observation to Control

Science Drives Functional Requirements

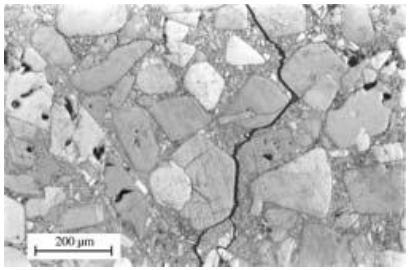
- **Control of Complex Materials and Chemical Processes**
  - Multifunctional materials: Correlated multiferroics, new photonic materials
  - Advanced metamaterials
  - Superconductivity



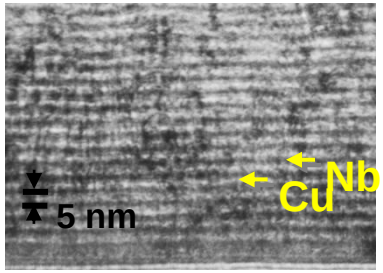
- **Energy Conversion and Storage**
  - Next generation photovoltaics with imbedded nanostructures
  - Advanced radiation detection
  - High density energy storage, super-capacitors, batteries



- **Dynamic Extremes**
  - Welds and casting
  - High Explosives dynamics – Equation of State

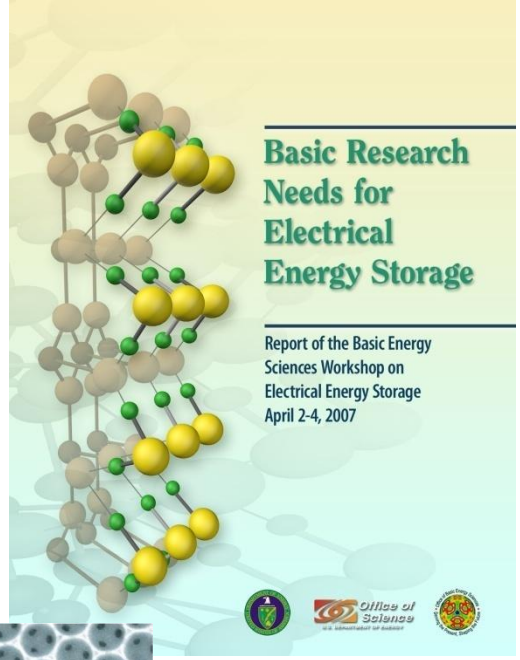


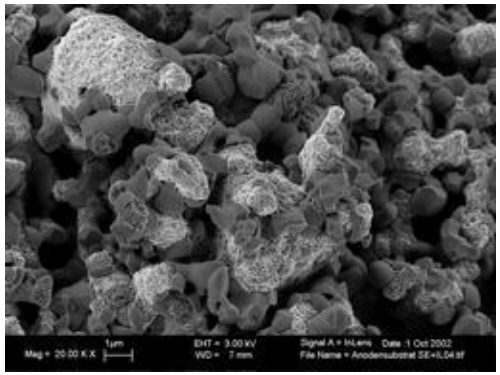
- **Radiation Extremes**
  - New radiation hard materials
  - Corrosion



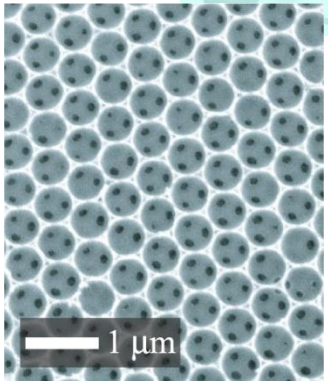
Energy Storage

- **Fundamental Issue**
  - How to efficiently store electrical energy in a high density form
- **Science - Control of electrons at interfaces**
- **Example - electrons to chemical bonds**
  - Efficiency at the anode (reducing the over potential)
  - Electrode/catalyst systems with no precious metals





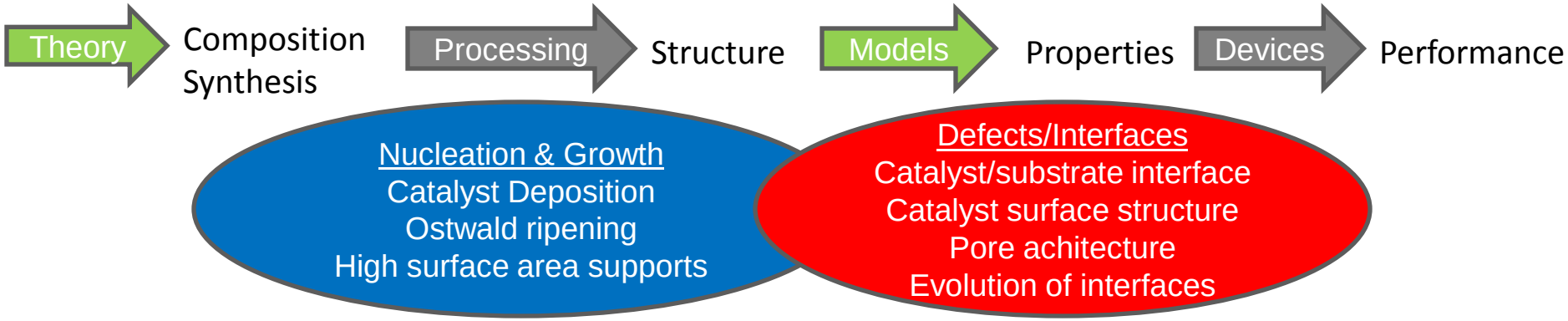
Observation



Control

Energy Storage

- Accelerate the discovery of new support/catalyst electrode systems for efficient high density storage of electrical energy as chemical fuels.
- Required to enable intermittent alternative energy sources such as wind and solar to expand significantly past the current 1-2 % contribution



Science based needs and gaps

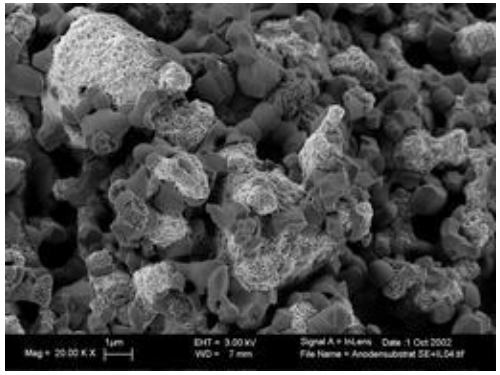
- **Needs**
  - Design principles to control electron transfer at interfaces
  - High surface area conducting architectures
  - Stable robust catalyst
  - Structures for efficient electron transfer and molecular diffusion
- **Performance Gaps**
  - Establishing the principles controlling electrode surfaces and electrode-electrolyte interfaces
  - Dynamic characterization of physical, chemical, and electrochemical properties
  - Theoretical modeling of electrode structure and design and electrochemical phenomena.
  - Synthesis of well defined conductive, porous electrode interfaces

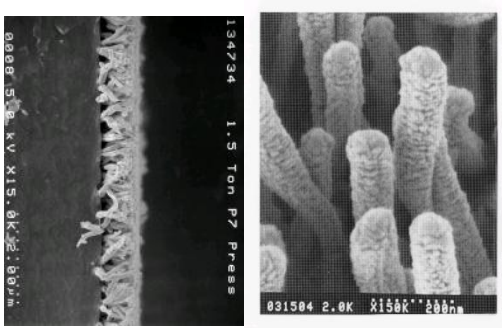
Energy Storage Functional Requirements

- **Extremes**
  - Repeated cycling in chemical pH extremes
  - Electric fields up to 1 V/nm
- **Measurements in situ (duringoperation, multiple measurements on the same sample)**
  - Measurement of structural changes of the catalyst 10 ns to second time scale (2-10 nm resolution)
  - Chemical changes at the solution interface 10 ns to second time scale
- **Synthesis and Characterization**
  - Well defined complex 3-dimensional conducting structures for electrodes: going beyond activated carbon
  - Controlled catalyst deposition
  - Ability to make a wide variety of conductive supports including oxides, nitrides, and carbides with high throughput – capacity to look at 100's to 1000's of samples quickly
  - Synthetic control band gaps –control of dopants and interfaces; going beyond Pt
  - Understanding of catalysis and electrolyte interactions in high electric fields up to 1V/nm
  - Characterization of integrated devices

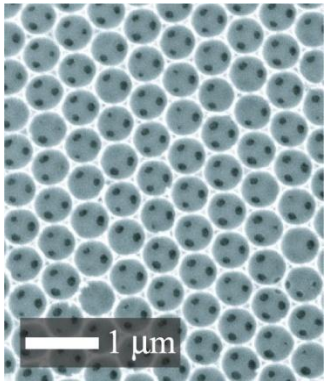
Challenges

- **Theory based design principles**
- **New porous supports/controlled catalyst deposition**
- **Catalyst evolution/surface chemistry**
- **Integrated device characterization and performance**

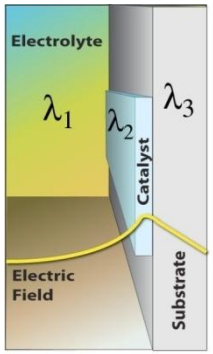




Perylene substrate by 3M



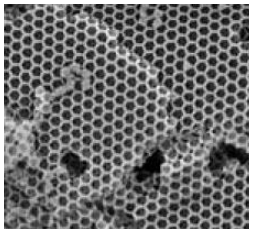
Energy Storage



Design of catalyst/substrate interface

1-D surface interfaces

Structure/Property Characterization at Multiple Scales

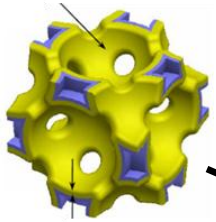


Inverse opals

High surface area electrodes

3-D architecture

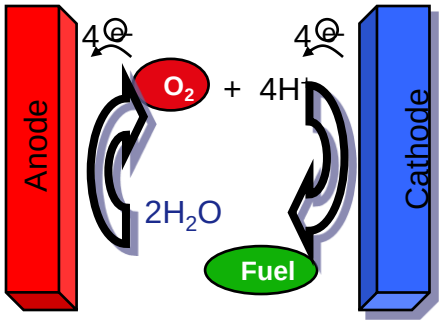
Interconnected cavities (<1 μm)



Bulk deeply buried interfaces

Selective deposition of catalytic sites

Integrated Device



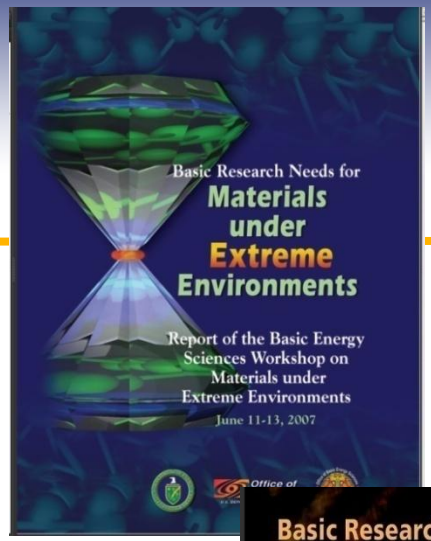
Atomic Understanding

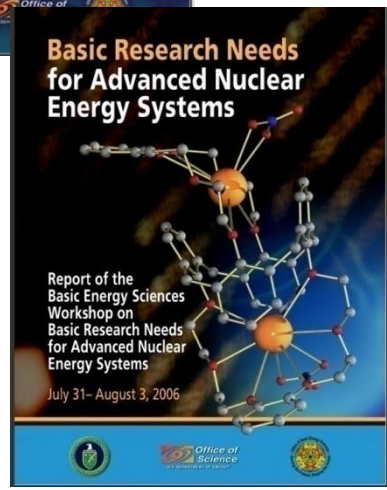
Control

Bulk Performance

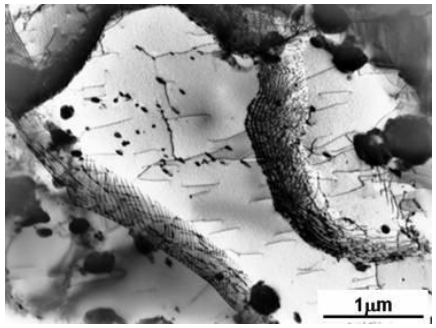
Slide 15

Materials in Radiation Extremes



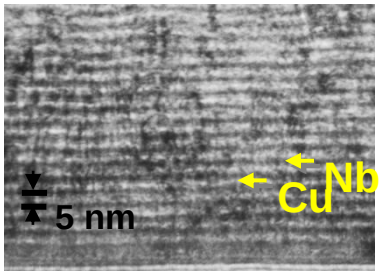


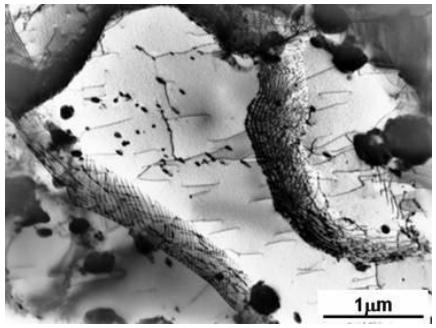
- **Materials stability under irradiation.**
- **Needs**
  - New radiation stable materials for fission reactors
  - First wall materials for fusion reactors and sensors
- **Example - Nanostructured ferritic alloys**
  - Accelerate the design of optimal structural materials for use in extreme irradiation environments using a requirements-driven methodology where material performance requirements drive process-aware synthesis.
  - Higher nuclear power plant efficiencies, greater burnup in nuclear reactors (need >40% for new reactors, currently at ~1% burnup), new class of materials for advanced fission and fusion reactors, longer lifetimes of structural materials in extreme conditions.



Functional Requirements

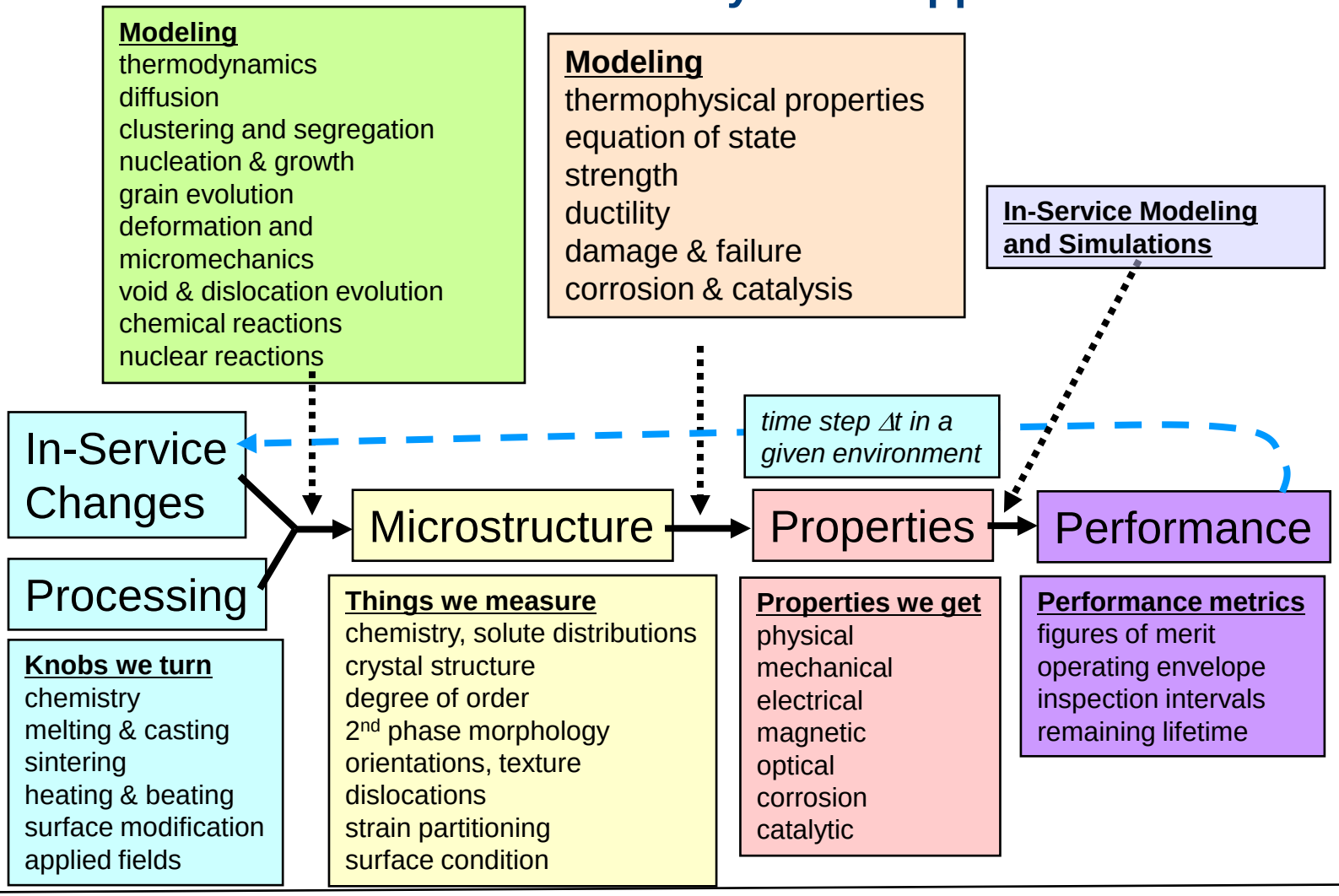
- Multi-scale modeling approaches that track defect evolution at interfaces in nanocomposites over length scales from atomic to macro, and time scale from ps to s
- Synthesis of bulk materials with well-controlled nano-structures
  - Metals, oxides, carbides, nitride samples with controlled composition and microstructures -
  - Synthesis of complex metals with precipitates
- Characterization of voids, defects and grain boundaries at atomic resolution
- Measurement of defect evolution in nanocomposites over length scales from atomic to macro, and time scale from ps to days
- 0 .0 1 to 10 DPA for volumetric swelling, irradiation creep, phase instability, hardening and embrittlement, thermal conductivity at temperatures up to 900 K





Process Aware Performance

Generalized Systems Approach



Frontier Experiments Define Functional Requirement Clusters

Frontier Challenges

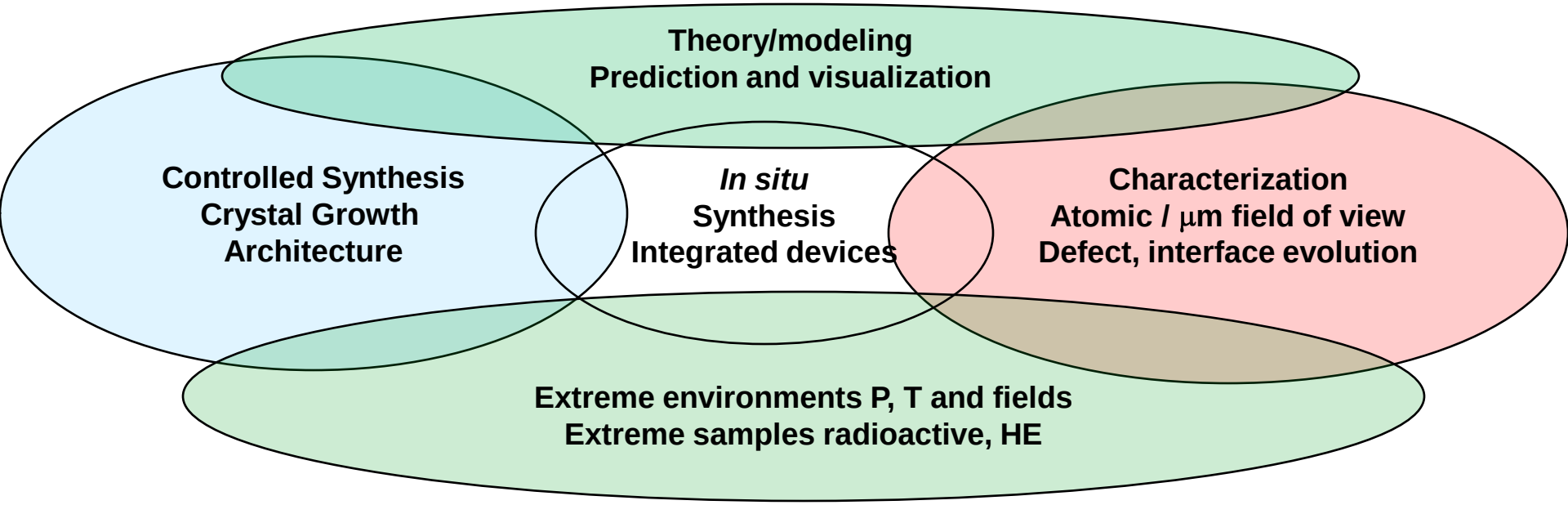
Energy Conversion and Storage

Radiation Extremes

Dynamic Extremes

Control of Complex Materials and Chemical Processes

Drive Functional Requirement Clusters



Define M4

FAB with complex materials

Evolution of defects/interfaces under irradiation

Crystal growth

Materials with controlled microstructure

Non Destructive 3-D Imaging

Dynamic structure-property relation

New Integrated LANL Facility

- M4 as Central User Interface – Gateway for MaRIE and to LANL
  - First comprehensive “control science for materials in extremes” user facility
  - Bridges broader LANL capabilities across scales: CINT← → M4 ← → Sigma/TA-55
- Quality
  - Reproducible well characterized materials
  - Infrastructure
- Accelerated Progress
  - Predictive theories to increase materials discovery success
  - Tailored materials by design for MPDH
  - Pre-irradiation of samples from FFMF
  - Integrated pre- and post-extreme measurement characterization
- Efficiency of Resources
  - Integration of resources and expertise due to LANL history and mission
  - Leverages CINT, Lujan, NHMFL, and IBML
  - Expertise with HE and radioactive materials

M4 4.0 Activities for Facility Definition

- ***Nucleation and growth***
  - Design of crystal growth facility
    - *Single crystal growth – traditional facilities for large crystals*
    - *In situ* characterization of crystal growth
    - Growth under pressure and in fields, and thin film techniques for meta stable materials
    - Single crystals with incorporated nanostructures
  - Materials with controlled microstructure.
    - Identify and design small scale casting facility to compliment bulk sigma activities.
    - Prioritize and compare top 1-3 *in situ* characterization tools (casting and welding as an example) with emphasis on understanding grain structure formation.
    - Evaluate potential techniques for process control of complex alloys including the incorporation of nanostructures

M4 4.0 Activities for Facility Definition

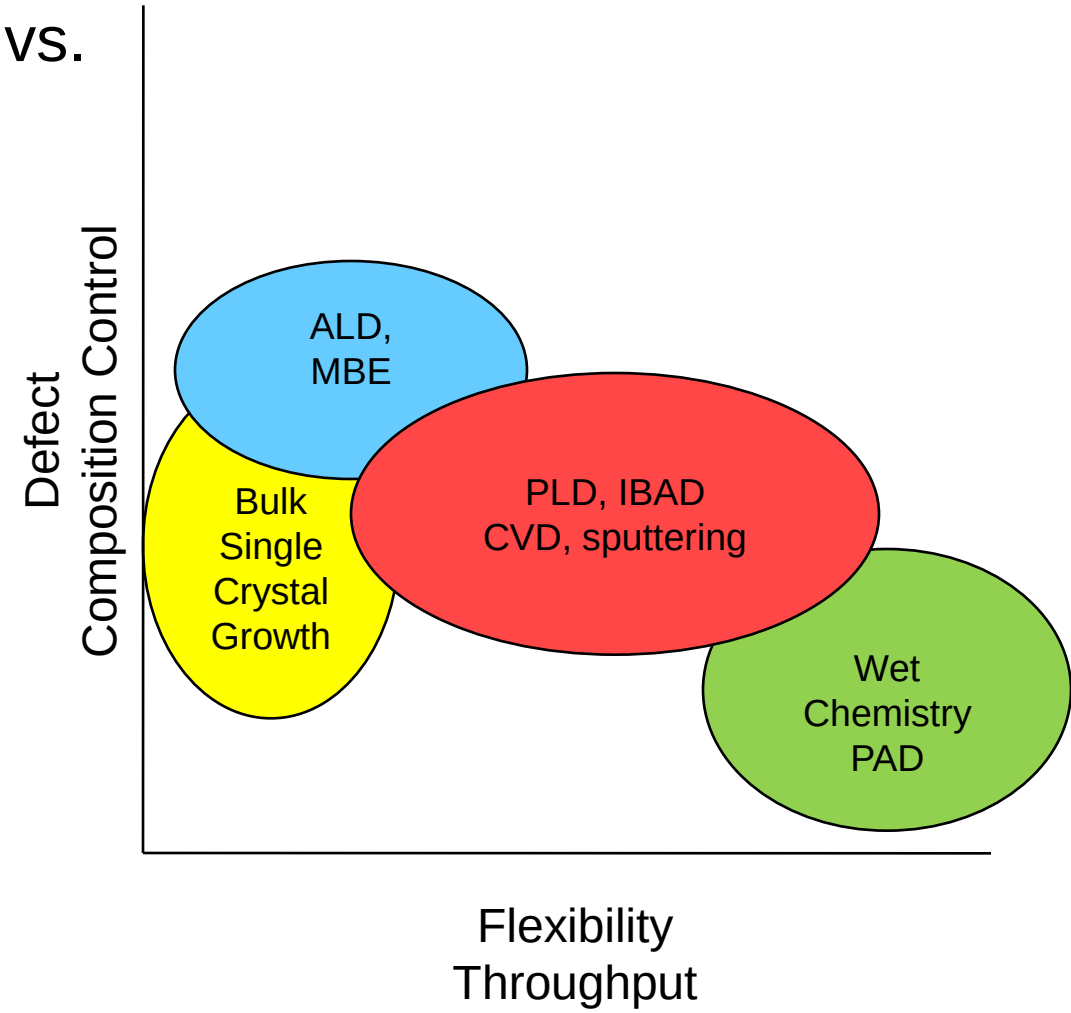
- **Complex Architectures**
  - *FAB with nontraditional materials*
    - FAB techniques with non semiconductor materials
    - Prioritize the key FAB techniques that are flexible enough for complex materials
  - 3-D Architecture
    - Composite materials
    - Routes to conductive 3-D porous materials

Synthesis – Crystal Growth

Defect/Composition control vs. Flexibility and Throughput

Composites

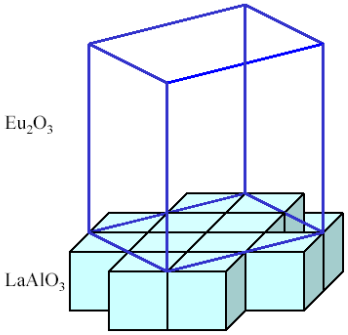
Bulk integration

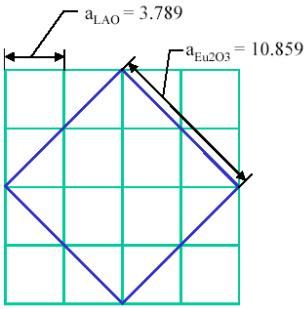


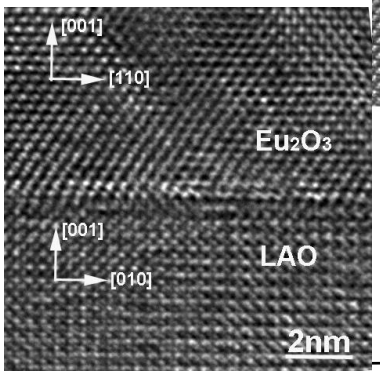
Crystal growth

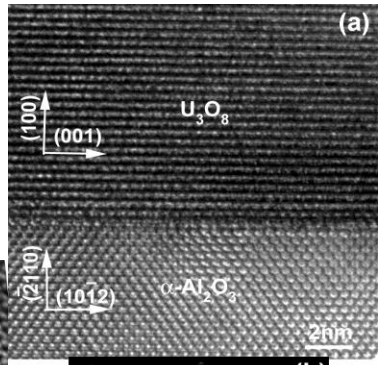
- **Current capabilities**
  - 3 Czochralski induction furnaces for oxide/intermetallics
  - 3 Czochralski tri-arc furnaces for intermetallics
  - Floating-zone furnaces (1 high-pressure) for oxide growth
  - Assorted cutting, grinding, polishing equipment
  - Powder XRD, 3 Laue XRD
  - Single crystal explosives growth [DE-9]
  - PLD, MBE, for oxide thin film growth , PAD for oxides, nitrides and carbides
  - *In situ* high temperature thin film X-Ray characterization (solution deposition currently)

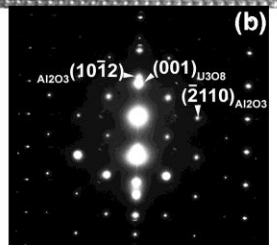
- **Future**
  - *In situ analysis of the growth surface*
  - *Rapid and high quality growth techniques*
  - *Crystal engineering of metastable materials*
  - *Composite materials*
    - *Growth of multiple phases*
    - *Incorporation of nanoparticles*

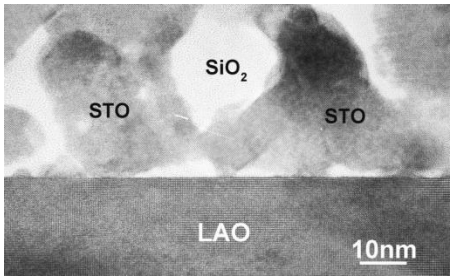


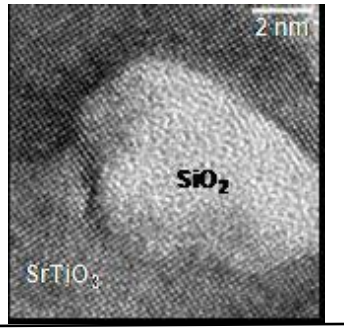








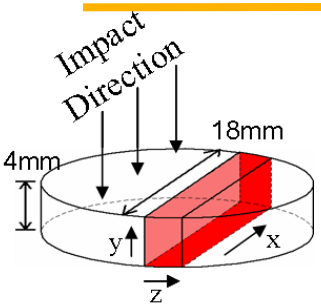




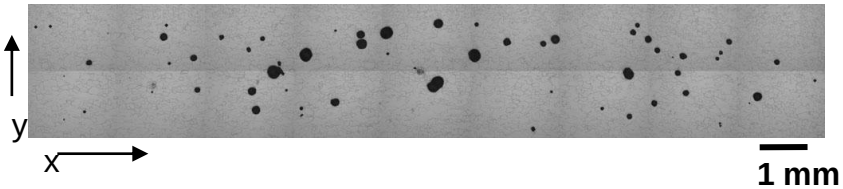
M4 4.0 Activities for Facility Definition

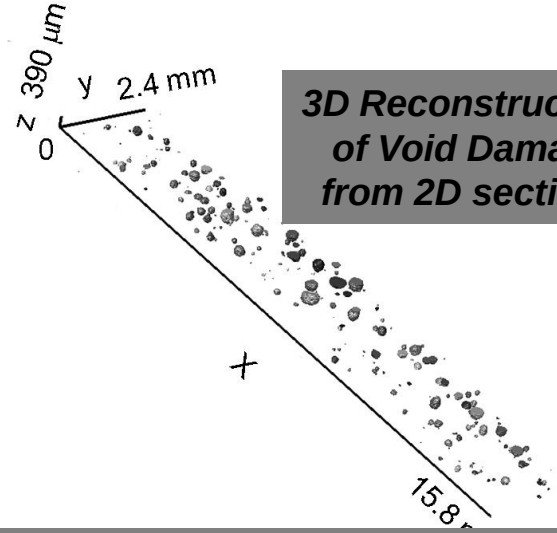
- **Advanced Characterization**
  - Non-destructive 3-D imaging
    - Use of electron, neutron and X-ray techniques to address voids, grain size/orientation,
    - Buried interfaces, composition inhomogeneity and structure/phase inhomogeneity.
    - Lab scale equipment housed in M4 focus on dynamic, *in situ*.
    - Mutiprobe SPM with spectroscopy
  - Multi probe characterization/spectroscopy tools at an MPDH and station
    - Identify current state of the art (APS end stations) and advances that will be developed soon (LCLS 2) .
    - Identify gaps that could be filled by multiprobe capabilities using the MPDH photon source and secondary probes at an M4 end station.
    - Example ARPES to obtain electronic density of states and structure information on a time scale relevant to phonon-phonon interactions or phonon-electron interactions

Characterization - 3-D Microstructure



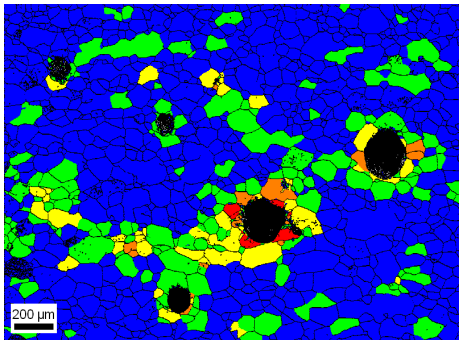
2D Montage of Spall Damage Field



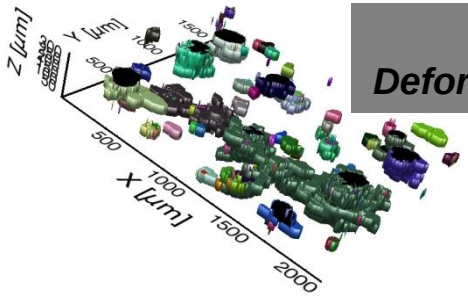


3D Reconstruction of Void Damage from 2D sections

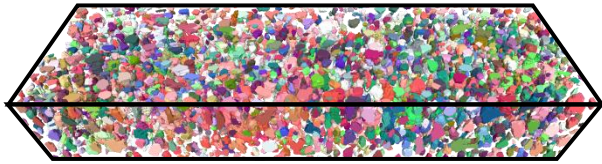
Mapping of Deformation Field from EBSD Orientation Data



Misorientation Range



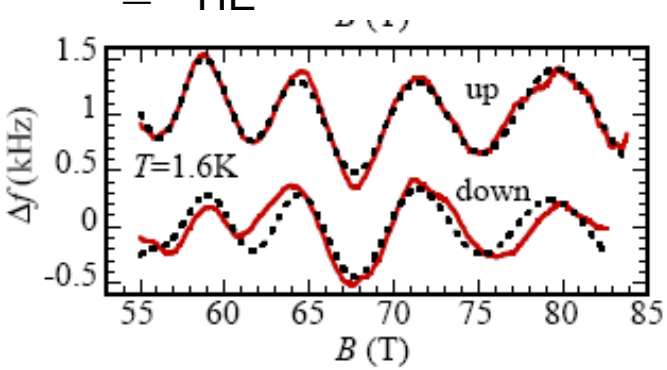
3D Reconstruction of Deformation Field and orientations

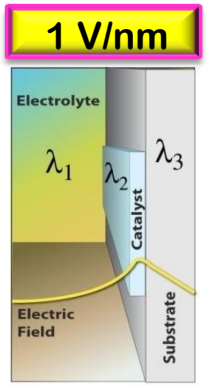


Non Destructive 3-D Tomography
- Higher Resolution
- Quantitative Information
  Structure, orientation, chemistry

M4 4.0 Activities for Facility Definition

- ***Characterization in extremes***
  - Radiation - design of ion beam lab
    - Identify and justify *in situ* characterization tools for ion beam stations.
    - Technical alternative analysis to show strengths and weaknesses of neutrons, X-rays, and electron microscopy.
    - Evaluation of lab scale end stations and photon source end stations such as APS, LCLS or MPDH.
  - Control of Extremes to Tune Properties
    - Extreme temperature for *in situ* measurements
    - Extremes for metastable states, pressure, IBAD
    - Extreme electric and magnetic fields
  - Facilities with authorization basis for hazardous materials
    - Radiological facility
    - HE





M4

- **Predictive Theory/Modeling**
  - Prediction of new classes of materials and determination of key processing parameters from IS&T data analysis
  - Theoretical Models that connect atomic scales to continuum
  - Validation and improvement of predictive fist principle theories
- **Flexible, integrated, solid state synthesis**
  - Single crystal growth including metastable materials
  - 3-D complex/composite architectures
  - Flexible fabrication capabilities for metals, oxides, nitrides, carbides
  - Characterization of nucleation and growth during synthesis
- **Characterization of structural and functional properties as they evolve**
  - Defect nucleation and growth evolution (e.g., TEM-scale resolution over mm dimensions – ability to index)
  - Static characterization of initial conditions
  - Ultrafast combined structure and function measurements of spin, charge, and lattice functionality
  - Evolution of defects and interfaces during function and in response to extreme environments
- **"Extreme environments" and "extreme materials"**
  - Irradiation environments (complementary to FFMF)
  - Chemical extremes for corrosion
  - Temperature and pressure, electrical and magnetic fields, dynamic compression environments
  - Radioactive and HE materials quantities (samples large enough to span 'micron frontier' within authorization basis)

Elegant experiments on Elegant (complex) materials

M4 Organizational Alternative Analysis Part I:

Existing Facility Options

Focus on integrated user-facilities co-located with photon or proton sources

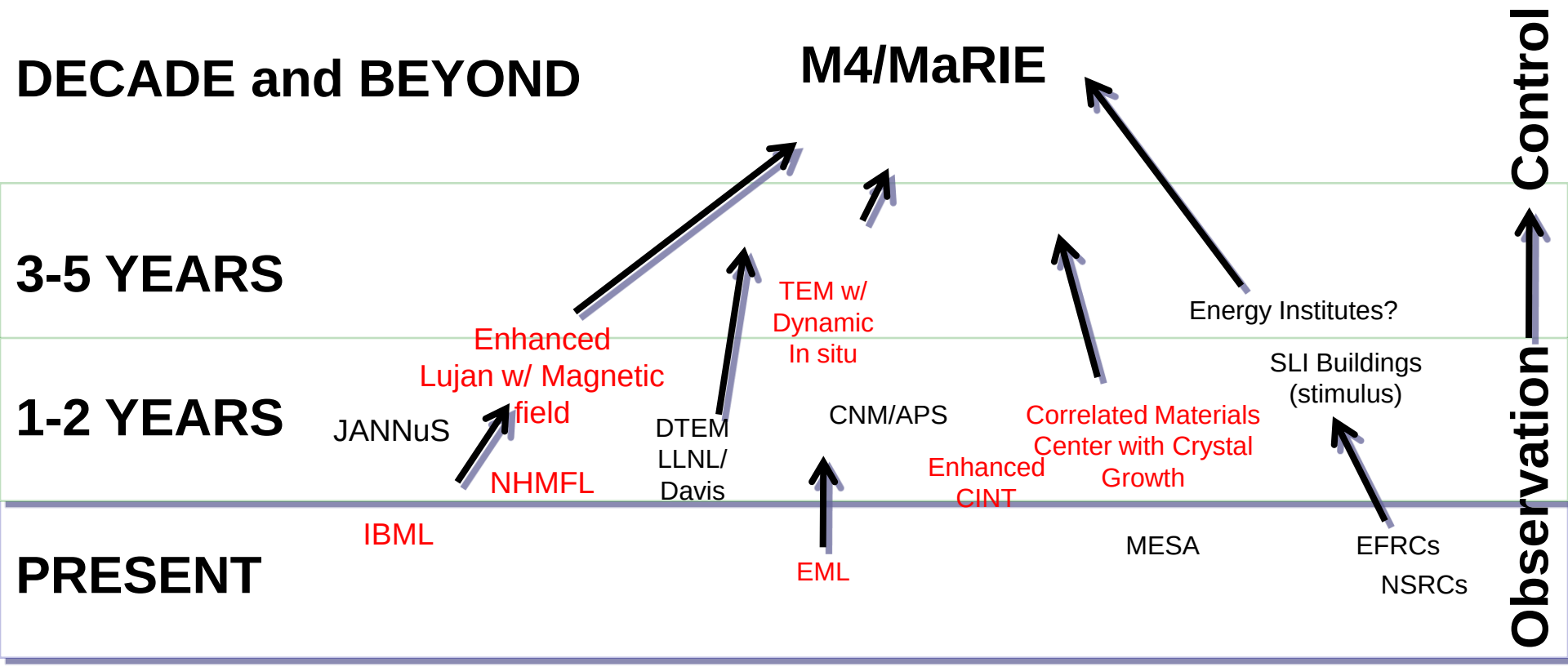
	EMSL PNNL	**MF** LBL	**CNMS** Oak Ridge	**CNM** Argonne	**CFN** BNL	**CINT-** LANL/ Sandia
Computational facilities						
Visualization capabilities						
Multi-scale theory capability						
Nanoparticles, wires, colloids						
Bulk materials/ nano features						
Single Crystal Growth						
Thin film deposition						
FAB						
3-D architectures						
***In situ* characterization of nucleation and growth**						
Device integration						

M4 Organizational Alternative Analysis Part I:

Existing Facility Options

	EMSL PNNL	**MF LBL**	**CNMS Oak Ridge**	**CNM Argonne**	**CFN BNL**	**CINT LANL/ Sandia**
Atomic characterization Defect detection	NMR, X-Ray	TEM,XPS	TEM, X-Ray	X-Ray	TEM, X-Ray	TEM, X-ray
Buried interfaces						
Surface Characterization						
***In situ* defect evolution**						
Ultrafast spectroscopy						
Ultrafast spectroscopy and structure						
Extreme environments	ion beam		temp	ion beam		NHMFL, ion beam
Beam Lines		ALS	SNS, HIFR	APS	NSLS	LANSCE

The Roadmap from CINT to MaRIE



RED = LANL
OTHER

Lab-Scale Extremes | In-Situ Characterization | Synthesis Capability | Integration (esp. theory)

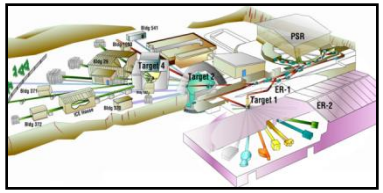
User Community Outreach

- **Progress to Date**
  - MaRIE workshop “Research Frontiers and Capability Gaps for Controlling and Designing Functional Materials” 1/20-1/22 2009 Workshop report.
  - 3.0 activities are defining internal and external working groups
  - Structural Materials Under Extreme Conditions

    Weds. July 29 – Fri. July 31 at the MSL

- **Road to MaRIE workshop 12/09**
  - *In situ* characterization of interfaces and defects is being assessed
  - Internal workshop on crystal growth – pushing the boundaries
  - Internal workshop on characterization – atomic resolution with large field of view

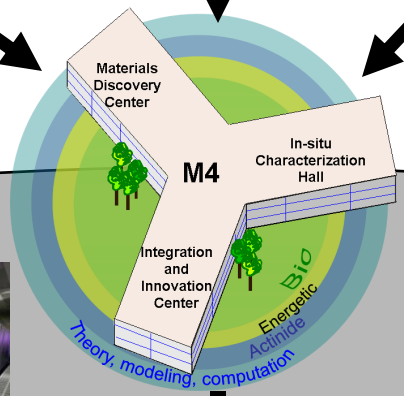
M4 MaRIE Integration



User Interface for MaRIE

FFMF-
- New materials by design
- Pre and post irradiation characterization
- Pre aging of samples

MPDH-
- Samples with controlled microstructure
- Complementary characterization tools



NHMFL

Roadrunner

Enhanced Lujan

CINT

Integrated Solid State Solutions

- Control of Complex Materials and Chemical Processes
- Energy Conversion and Storage
- Dynamic Extremes
- Radiation Extremes