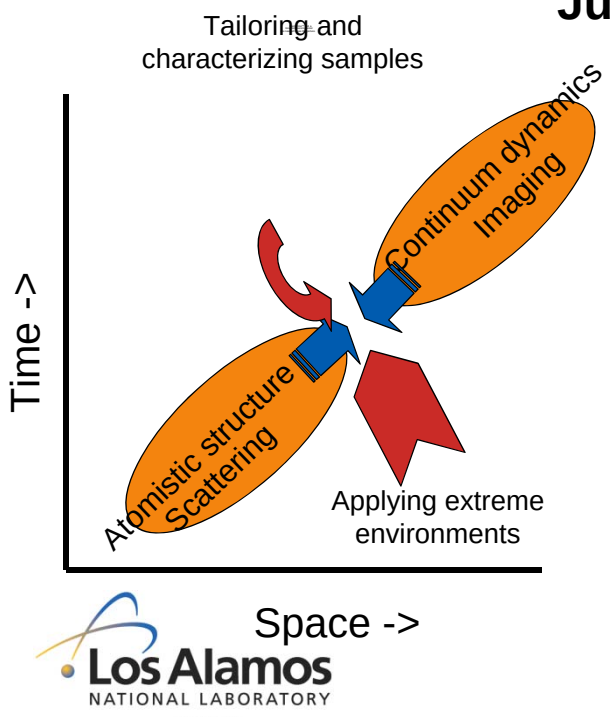
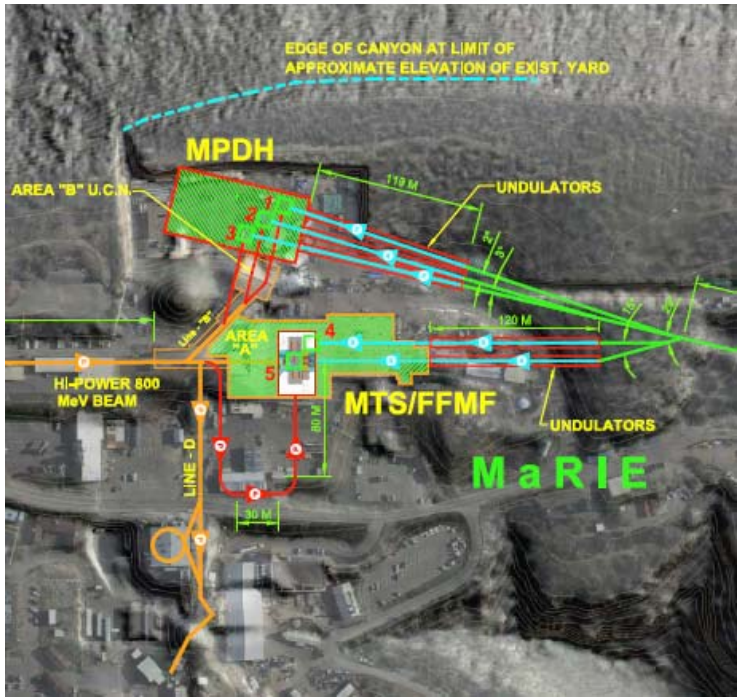
The MaRIE Multi-Probe Diagnostic Hall

Cris W. Barnes

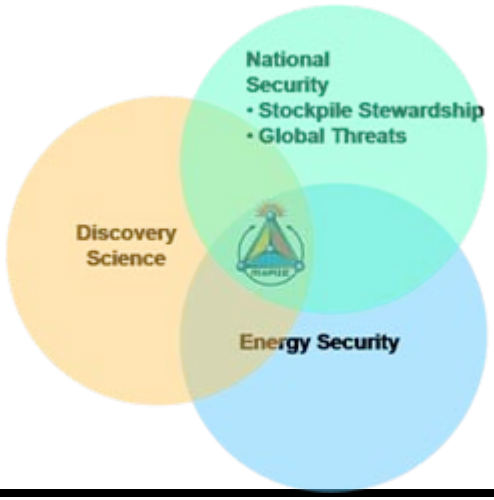
Los Alamos National Laboratory

June 11, 2009



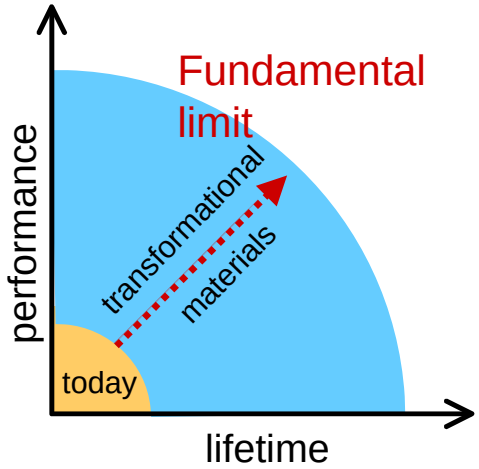


MaRIE: Materials R&D Capability for 21st Century National Security and Energy Security



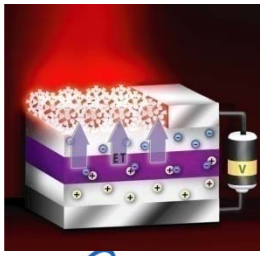
MaRIE addresses materials challenges across missions

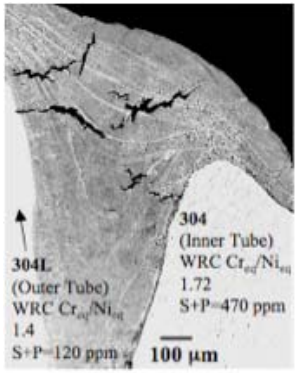
MaRIE enables the transition from "observation" to "control"



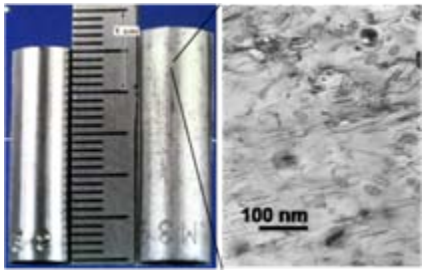
MaRIE will transform the science of microstructure, interfaces, and defects

Next-generation solar cell architecture



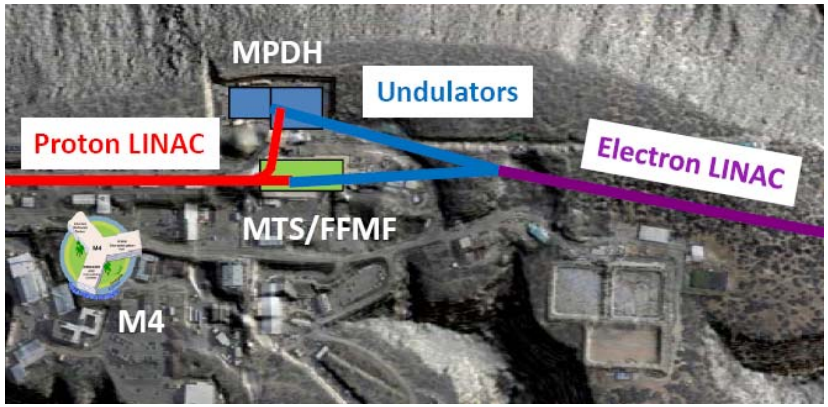


Cracks in welds



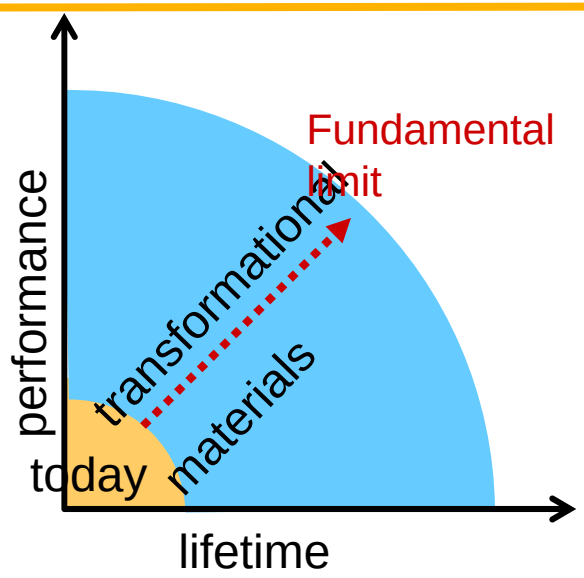
Radiation-induced swelling

MaRIE provides tools for transformational materials performance in extremes



The transition from "observation & validation" to "prediction & control" is a central mission challenge AND the frontier of materials research

Achieve Transformational Materials Performance
-Solutions require unprecedented control of defects and interfaces



Through Predictive Multi-scale Understanding
-Perform experiments with unprecedented spectral, temporal, and spatial resolution in previously un-accessed extremes

with an emphasis on Radiation-Matter Interactions
-Nuclear is special for LANL and for the world

-LANSCE is key to our uniqueness in materials-centric national security science

MaRIE will be the first capability with unique co-located tools necessary to realize transformational advances in materials performance in extremes

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Frontier Experiments define Functional Requirement Clusters

Frontier Experiments

Compression Dynamics	Structural Materials in Dynamic Extremes	Control of Complex Materials and Chemical Processes	Turbulent flows	Warm Dense Matter

Drive Functional Requirements

Defect and Dislocation Dynamics; Dynamic performance of bulk material	*In-situ density, volume, macrostructure and cracks*	*Ultrafast pump-probe; swept spectroscopy; nano-imaging*	*Multiple measurements over longer times*	*Intense ion beams*

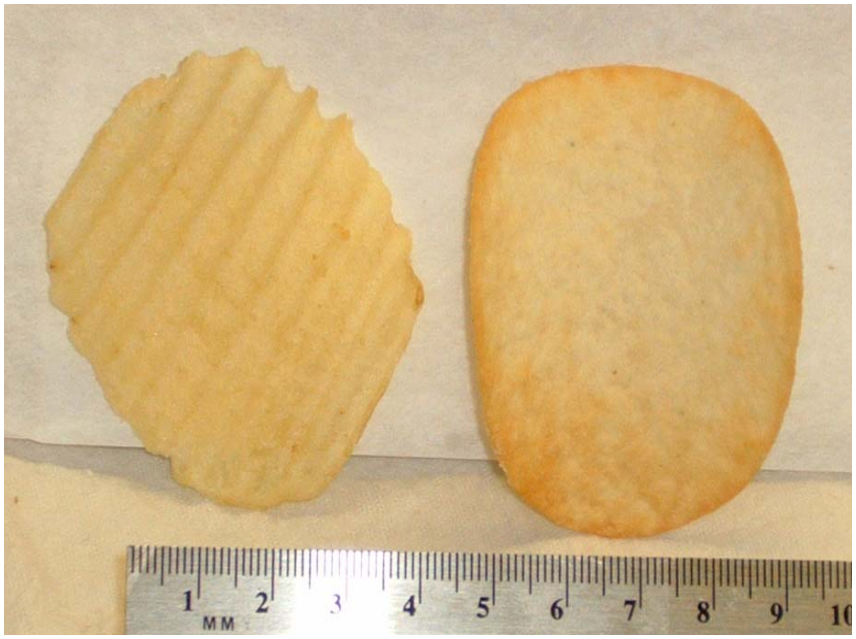
Which Lead to Technical Alternatives

High-energy coherent x-ray imaging; dynamic electron microscopy; variable strain-rate drive; multiple surface diagnostics	Proton radiography; high-energy transient x-ray diffraction	Next generation temporal control for x-rays; optical ultra-fast lasers	Limits on duty cycle; phase contrast imaging	High-intensity ultra-fast laser system

Predictive capability of materials requires "process-aware" models

- Properties depend on structure, chemistry

Wrought Cast



- Processing can change structure

Processing controls structure + chemistry = properties = performance

Another example of “Process-Aware”

Feed Material + Processing Differences

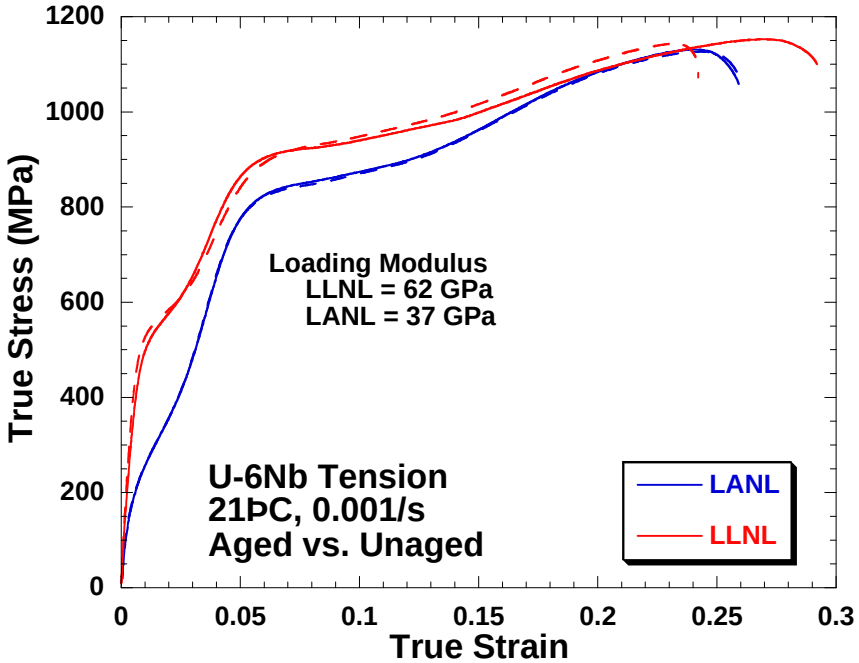
Processing + Aging

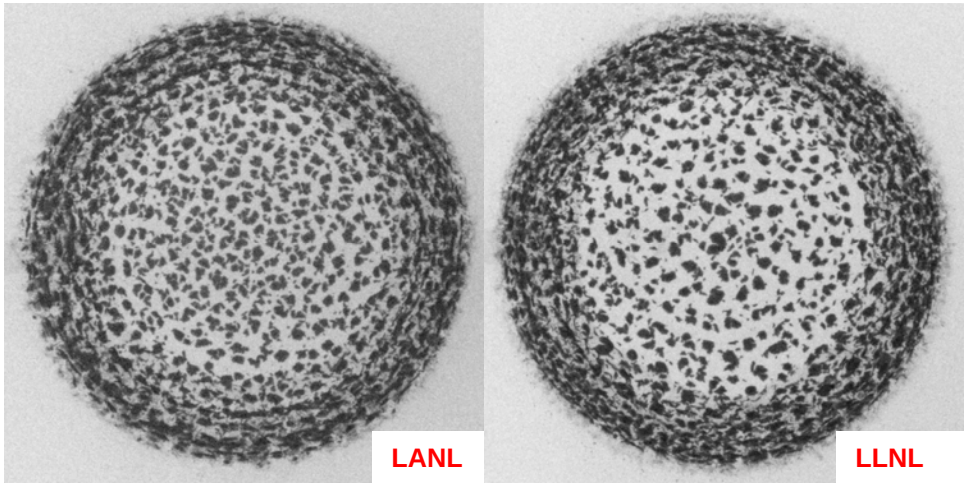
Processing controls structure + chemistry = properties = performance

Mechanical behavior and HE-driven fragmentation of U-6Nb show strong influence of metallurgical state

LANL = Solution treated / Quenched

LLNL = Solution treated / Quenched + Aged



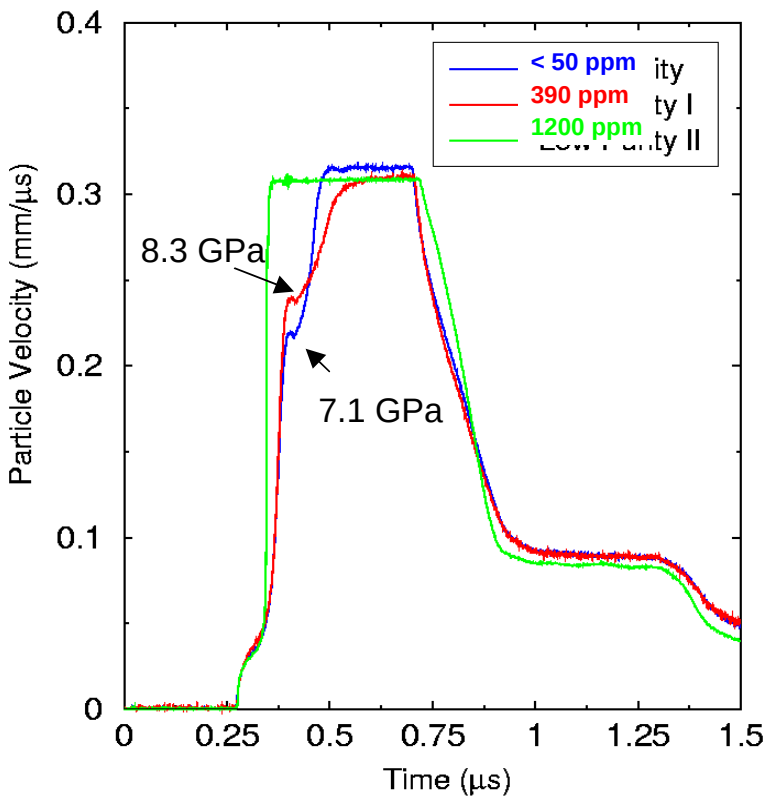


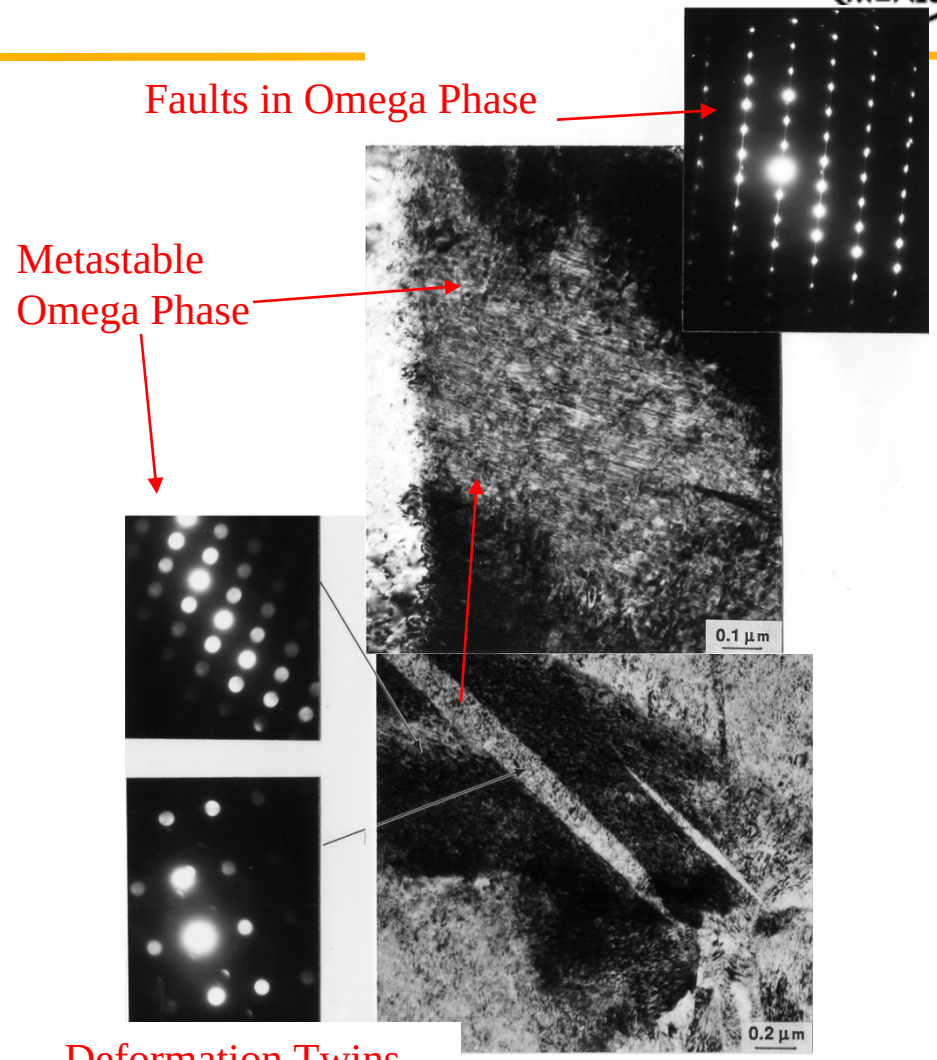
Quantitative analysis reveals 6σ difference in open area between images

Process-aware understanding of materials performance is lacking

Shock-induced phase transitions - spatially complex processes with strong materials sensitivities

Oxygen content suppresses the $\alpha-\omega$ phase transition in Zr



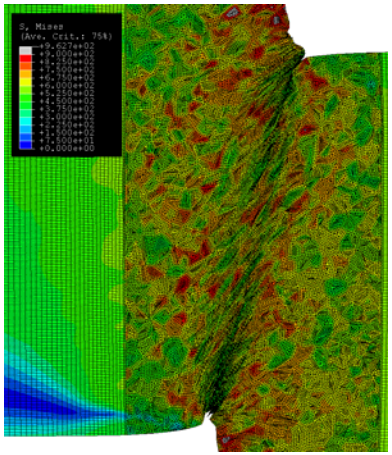


Predictive models must acknowledge that solids are an assembly of crystals that deform according to their local state of stress

Overview - Materials Models Used at LANL

Macro-Mech.

Numerous constitutive models

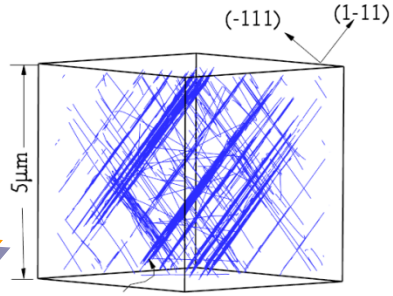


Polycrystal

Visco-plastic self consistent

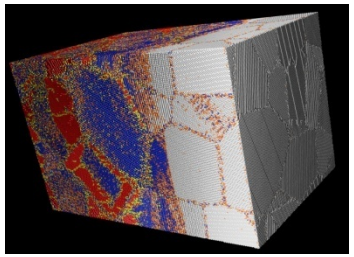
Single Crystal

G-L
Disl. Dyn. code



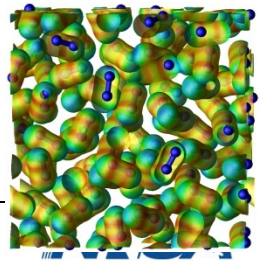
Molecular Dynamics

SPaSM
LAMMPS



Quantum Mechanics

VASP
MondoSCF



MaRIE data here

Physics Today, February 2009

Coherent X-Ray Diffractive Imaging

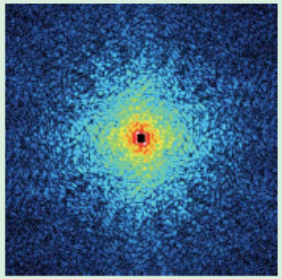
- **Detectors that record x-ray diffraction patterns can't measure the phases in a continuous diffraction pattern. When the pattern is sampled at a fine enough spatial frequency, the phases can be extracted and the object structure derived by using a computer algorithm that iterates back and forth between real and reciprocal space.**

- **Key leaders of this technique are Henry Chapman (Centre for FEL science at DESY[1]), Franz Pfeiffer[2] (formerly PSI, now at TU Munich), and Jianwei Miao[3] (UCLA)**

1. http://hasylab.desy.de/science/cfel/index_eng.html
2. http://people.web.psi.ch/pfeiffer/
3. http://www.physics.ucla.edu/research/imaging/index.htm

A three-dimensional x-ray image of a single pair of human chromosomes

X-ray crystallography routinely yields the structures of proteins and other biomolecules with 0.1-nm resolution. But how does one take a similarly detailed look at something far bigger—a chromosome, say, or a cell nucleus? Such objects don't crystallize because no two individuals have the same shape. And at a micron or so in size, they're too thick for electron microscopy. Electrons either get stuck in the object or scatter too many times on their way out to form a clear image.



X rays, of course, pass through whole animals, not just single cells. For the past decade or so, groups around the world have been developing a technique called x-ray diffraction microscopy for imaging single, uncrystallized samples.

The first biological application of XDM appeared in 2003. Jianwei Miao, who's now at UCLA, and his collaborators obtained two-dimensional images of single *Escherichia coli* bacteria with 30-nm resolution.[1] To boost the scattered signal, Miao had to stain the bacteria beforehand with a manganese salt. Staining doesn't damage the sample, but it biases the image because only certain amino acids stain readily. Two years later David Shapiro of Stony Brook University and his collaborators demonstrated that XDM could yield 2D images of single yeast cells without staining.[2]

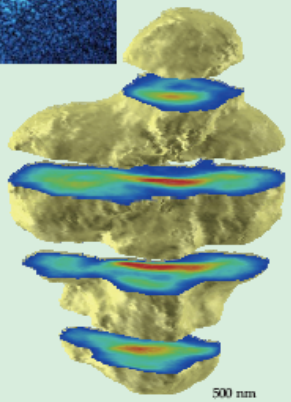
Now, extending XDM's steady progress, Yoshinori Nishino of the SPring-8 synchrotron in Sayo, Japan, and his collaborators have created 3D images of pairs of unstained human chromosomes.[3] The result not only represents a new XDM milestone, it's also biologically significant: Nishino's images reveal that a human chromosome in its most compact state has a dense axial backbone.

In XDM, coherent, monochromatic x rays scatter off electrons in the sample's constituent atoms to form a continuous diffraction pattern, like the one shown above on the left. The pattern's features embody the length scales of substructures present in the sample but not the substructures' locations. As in all diffractive imaging, obtaining those locations—and therefore deriving the structure itself—requires knowing the phases of the scattered x rays.

The detectors that record x-ray diffraction patterns can't measure those phases directly, a shortcoming that gives rise to crystallography's famous phase problem. In 1952 David Sayre suggested a solution that would later be applied to XDM. The all-important phases, Sayre realized, are not lost, but reside in the continuous diffraction pattern. When the pattern is sampled at a fine enough spatial frequency, the phases can be extracted and the structure derived by using a computer algorithm that iterates back and forth between real and reciprocal space. The first demonstration of XDM was performed by Miao and colleagues in 1999; it used an algorithm devised in 1978 by James Fienup (see Physics Today, October 1999, page 9).

Human chromosomes provide an apt testbed for XDM. At its most basic structural level, a chromosome consists of a strand of negatively charged DNA. To hold the information needed to encode a genome, the strand would stretch to about a meter in length. But to fit inside a cell's nucleus and to enable gene transcription to proceed in an orderly way, DNA must be compacted. Positively charged proteins called histones help DNA fold and coil into a hierarchy of ever-denser structures that culminates in mitotic chromosomes, the paired form that appears during cell division. A mitotic chromosome is typically a few microns long. Pairs of them can be seen with an optical microscope. Their surface can be mapped with atomic force microscopy, but their internal structure is not fully known.

The figure below shows the SPring-8 team's 3D image of a pair of mitotic human chromosomes joined at their middles in an X-like shape. The arms of the chromosomes have dense, axial backbones, suggesting a high level of packing.



In principle, the resolution of XDM is limited only by the x rays' wavelength. In practice, x rays would damage and deform the sample long before the signal was strong enough to yield the limiting resolution. The SPring-8 team obtained the same 30 nm resolution in 2D—as Miao and Shapiro did for their pioneering images. But for the 3D image, which required combining exposures made at 38 different viewing angles, the resolution was 120 nm.

Radiation damage is unavoidable but it can be mitigated. Encasing a sample in ice not only boosts radiation resistance, it also preserves—or comes close to preserving—the sample's natural, hydrated state. Freezing a sample risks introducing spurious signals from ice crystallites, but it could yield a 3D resolution of 10 nm.

Radiation damage can also be made irrelevant. In 2006, Henry Chapman, Janos Hajdu, and their collaborators resolved a single object with a free-electron laser. The FEL's intense pulse destroyed the sample, but not before photons had scattered off the atoms and carried away structure-determining information. (See Physics Today, January 2007, page 19.) Chapman and Hajdu's demonstration used extreme UV photons, which could not have resolved a chromosome. That barrier will fall when SLAC's Linac Coherent Light Source and other x-ray FELs come on line next year.

Charles Day

References

1. J. Miao et al., *Proc. Natl. Acad. Sci. USA* **100**, 110 (2003).
2. D. Shapiro et al., *Proc. Natl. Acad. Sci. USA* **102**, 15343 (2005).
3. Y. Nishino et al., *Phys. Rev. Lett.* **102**, 018101 (2009).

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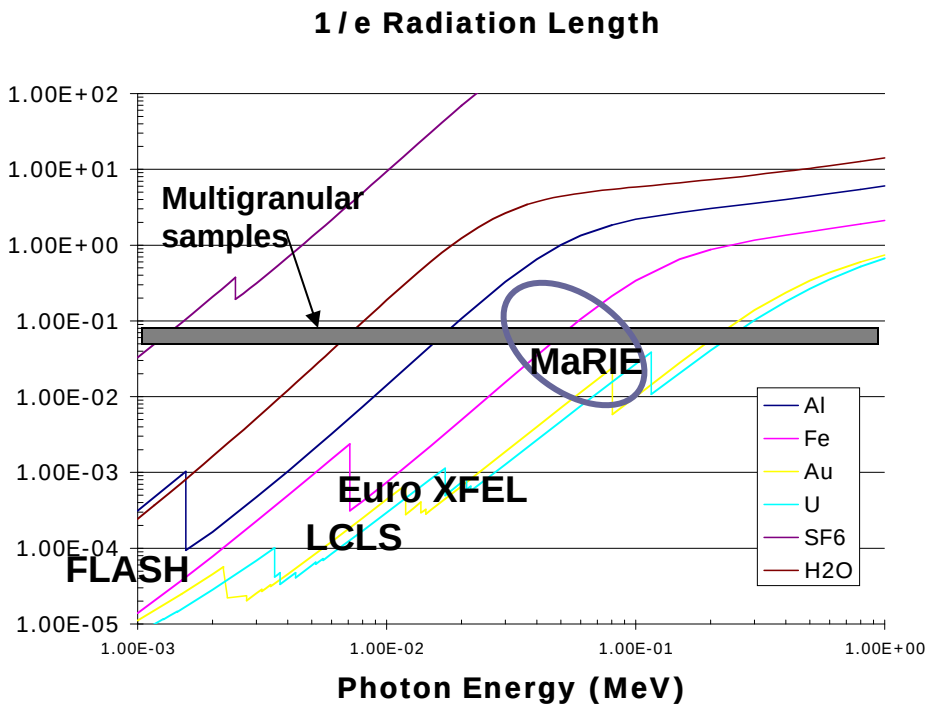
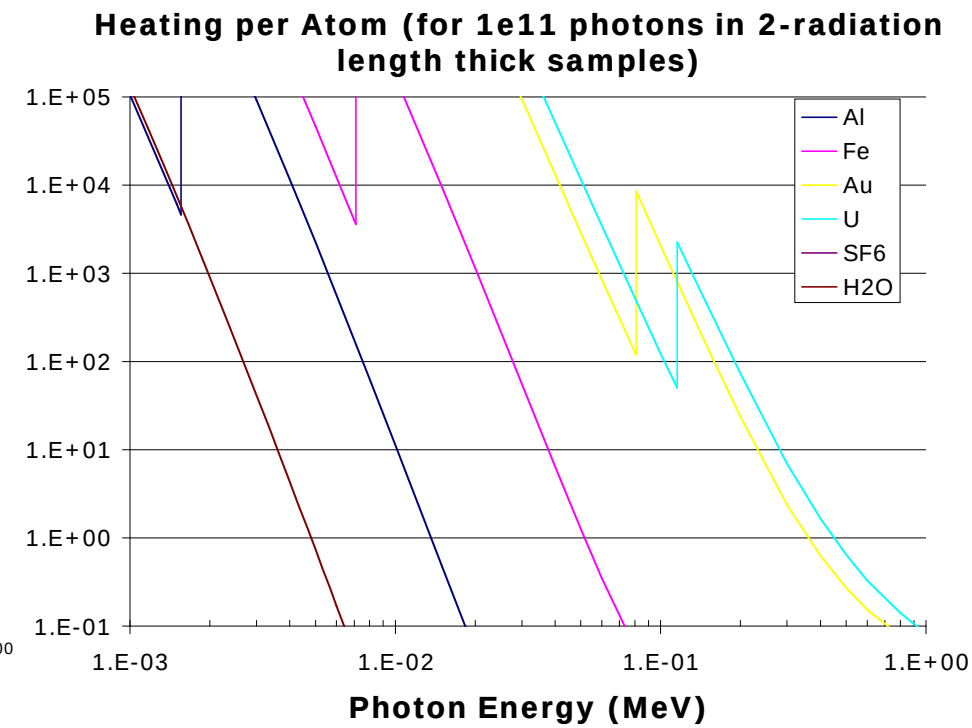
A high-energy-photon (50-115 keV) X-ray Free Electron Laser allows multigranular sample penetration and multipulse dynamics without significant sample perturbation



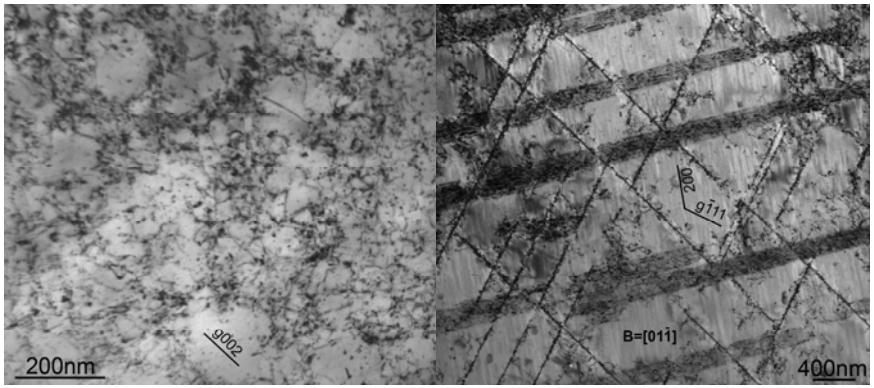


Meanwhile, proton and/or electron microscopy can provide absolute density and velocities through the sample volume.

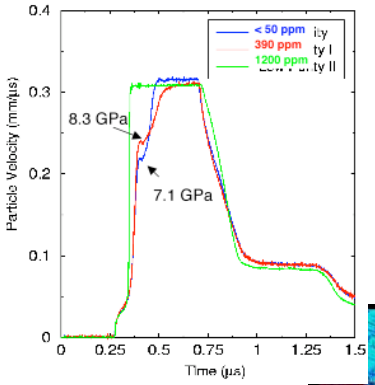
Compression dynamics

Defect and Dislocation Dynamics; Dynamic performance of bulk material

- **Shock dynamics**

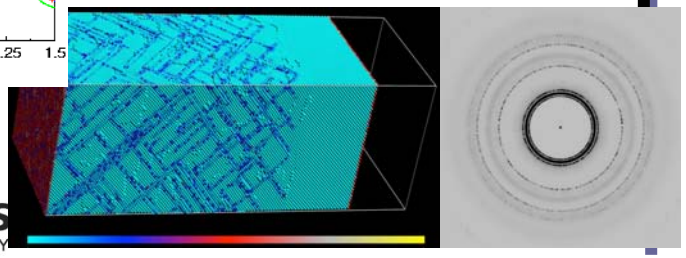


Shock effect on different crystal orientations



Impurities affecting phase transitions

Developing quantitative analysis

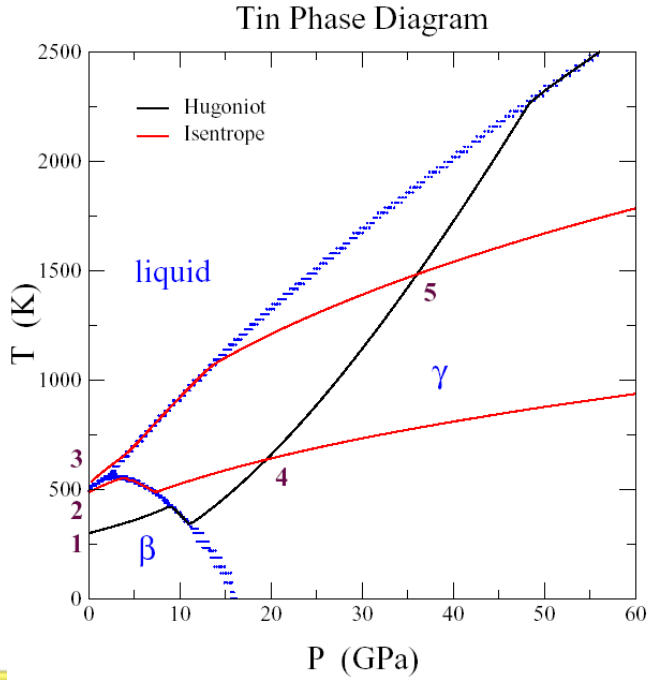


- **Path Dependence of Strength**

Use two paths to access the same state.

- 1 => 4, Shock loading with **high** strain rate and **solid-solid** phase transition.
- 2 => 4, Isentropic loading with **low**, variable strain rate and **solid-solid** transition.

Measure micro/meso-structure *in situ*

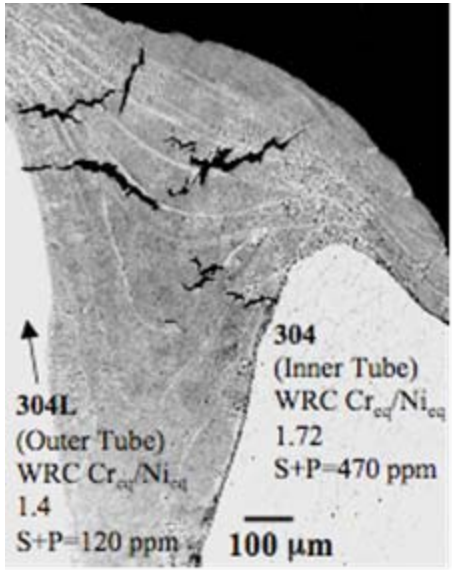


Predictive capability for safety and surety improvements possible

Structural Materials in Dynamic Extremes

In-situ density, volume, macrostructure and cracks

- **Welding**



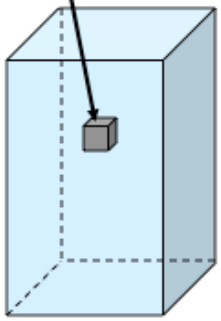
- **In-situ Radiation Damage**

See talk next week by Mark Bourke!!

- **Phase Transformations**

At every point in space & time we want to know:
- crystal structure
- composition
- degree of order (chemical, magnetic, electric polarization)

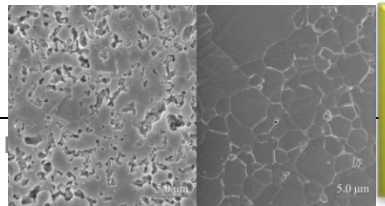
Also want to map process variables:
- stress
- strain
- temperature
- fluid velocity



- **Armor**

Spatial resolution = maximum spacing between neighboring volume elements

Temporal resolution = maximum time gap between successive measurements (conditions probed) during an in-situ run

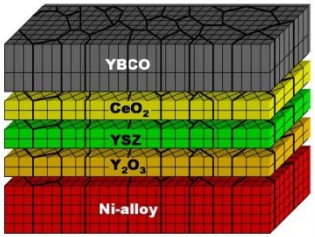


Controlled functionality of materials certified without test

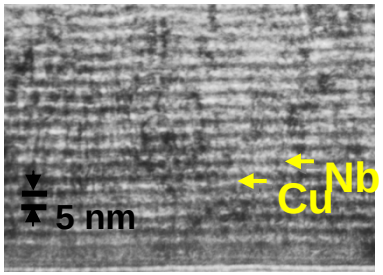
Control of Complex Materials and Chemical Processes

Ultrafast pump-probe; swept spectroscopy; nano-imaging

- **Emergent Phenomena**
  - Correlated Multi-ferroics

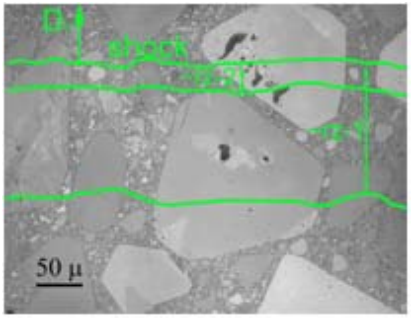


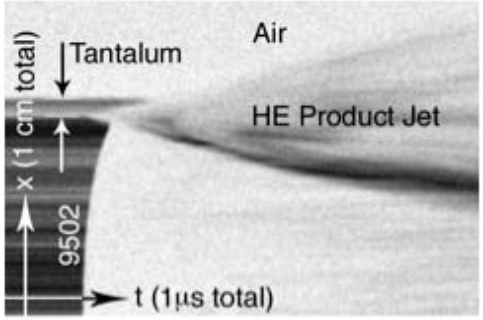
  - Radiation Resistant layered materials

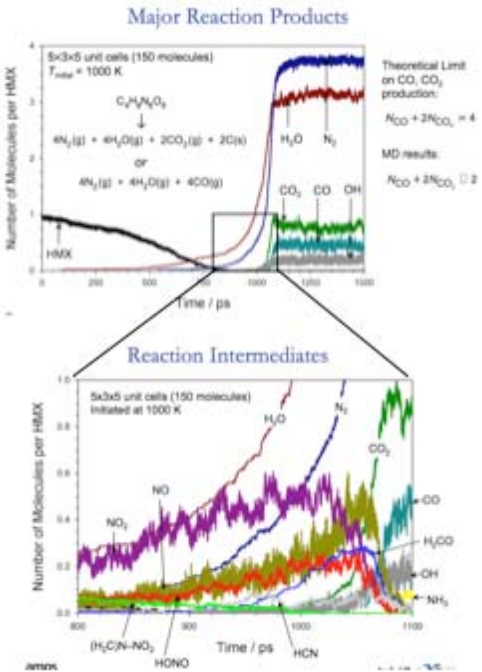


Advanced Energy systems or Energetic materials

- **High Explosives Burn and Chemistry**



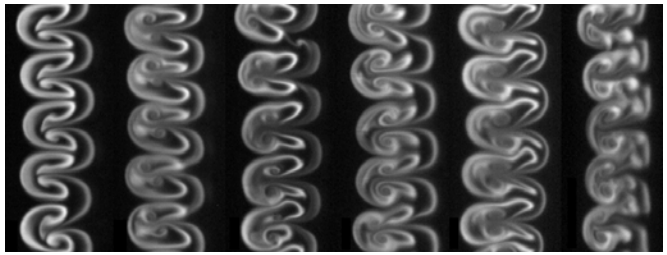


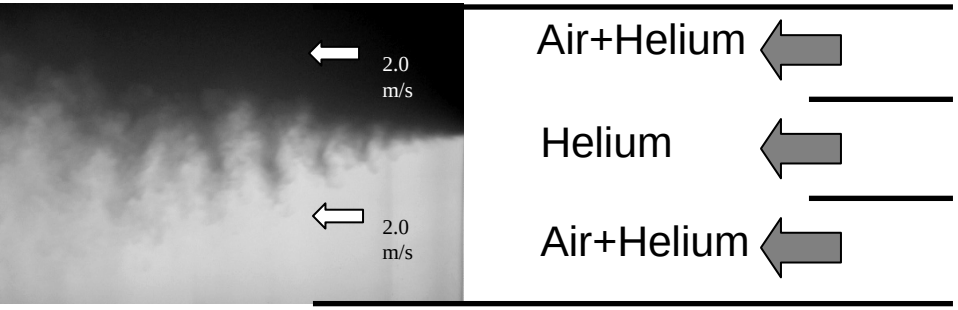


Turbulent Flows

Multiple measurements over longer times

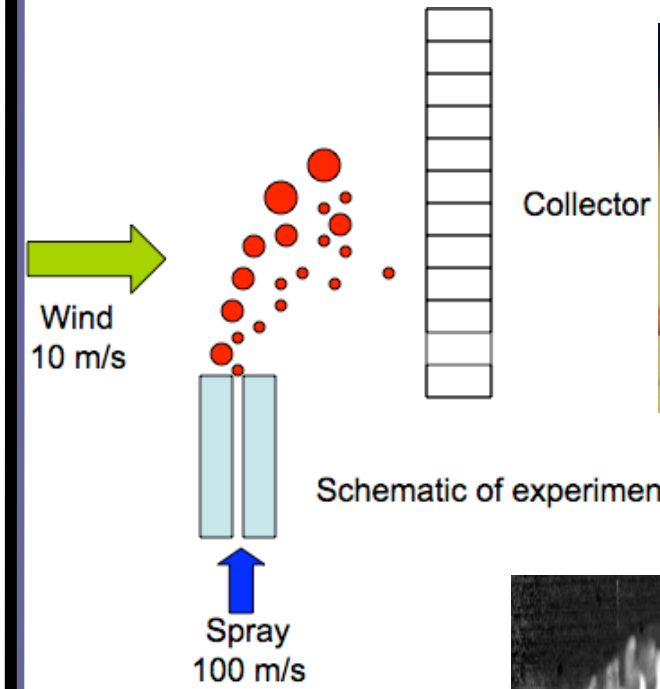
- **Rayleigh-Taylor or Richtmyer-Meshkov in water tank or gas channel**



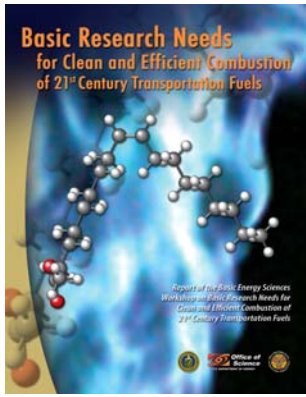


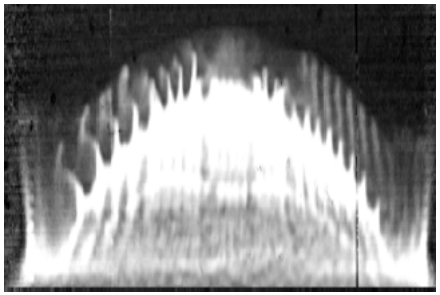
Validated climate models or boost predictive capability

- **Spray and Combustion**
  - MaRIE can resolve the dynamics of "fuel" spray formation



Schematic of experiment





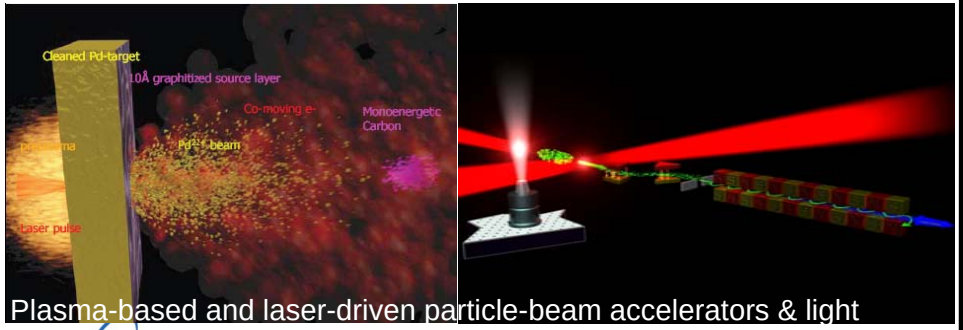
Related to ejecta measurements and boost science

Warm and Hot Dense Plasmas

Intense ion beams

Plasma tools will be used:

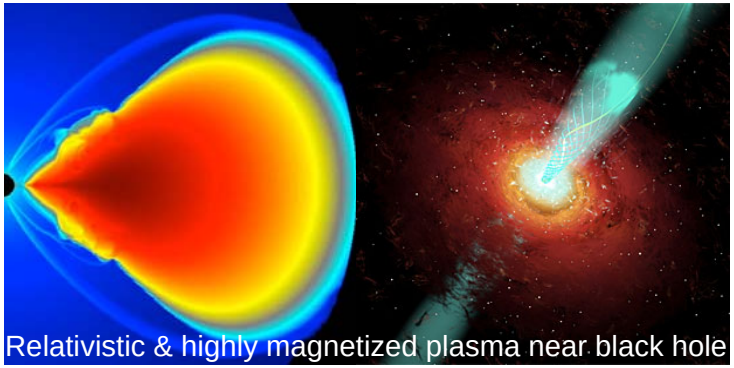
- Ultrafast optical lasers for pump-probe
- Long-pulse drive lasers for variable strain compression
- Combined high-intensity systems to provide pulsed ion and neutron sources
- As design advances, plasma acceleration techniques may play a role



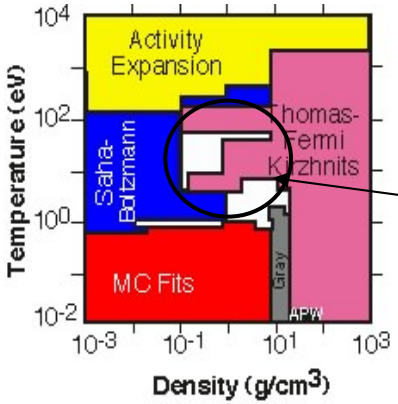
Plasma-based and laser-driven particle-beam accelerators & light

Warm and Hot Dense Matter

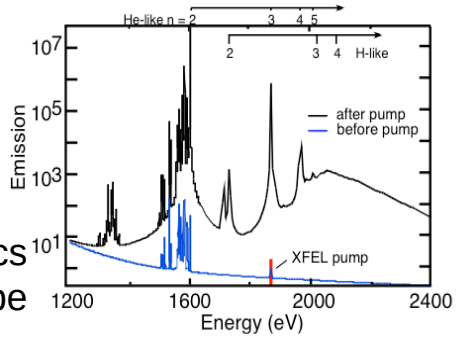
Warm-dense matter near core of Jupiter



Relativistic & highly magnetized plasma near black hole



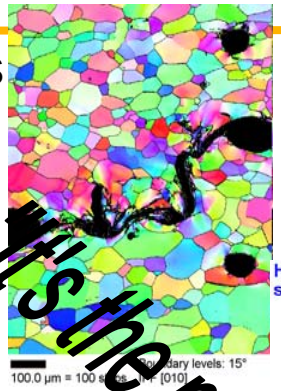
Warm Dense Matter



Extreme atomic physics using pump-probe

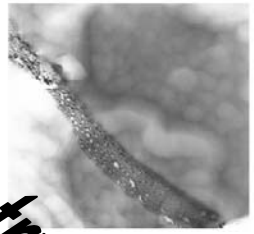
We need MaRIE to bridge the "micron gap"

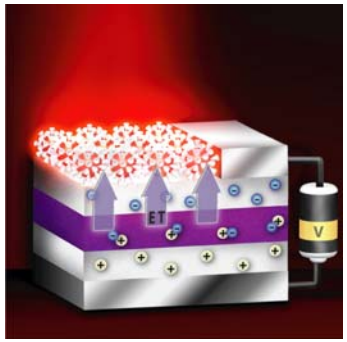
- ~ 1 μm scale (and time scale ~sound transit) represents an experimental *and* theoretical frontier, where discovery science and predictive validation meet
- We will bridge the atomic scale/molecular dynamics studies and continuum models/integrated tests.
  - Defect consequences and microstructure interactions that drive materials strength and damage evolution
  - Translation of unit-scale emergent functionality to device realization/interface phenomena

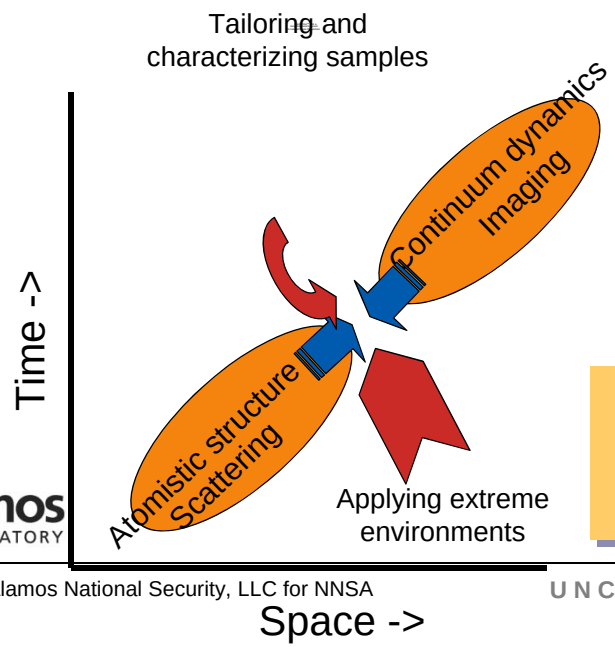


"It's the microstructure, ..."

He bubbles on grain boundaries can cause severe embrittlement at high temperatures







MaRIE will provide unique capabilities for unraveling micron-scale interactions, bridging the regime between imaging and scattering

Frontier Experiments define Functional Requirement Clusters

Frontier Experiments

Compression Dynamics	Structural Materials in Dynamic Extremes	Control of Complex Materials and Chemical Processes	Turbulent flows	Warm Dense Matter

Drive Functional Requirements

Defect and Dislocation Dynamics; Dynamic performance of bulk material	*In-situ density, volume, macrostructure and cracks*	*Ultrafast pump-probe; swept spectroscopy; nano-imaging*	*Multiple measurements over longer times*	*Intense ion beams*

Which Lead to Technical Alternatives

High-energy coherent x-ray imaging; dynamic electron microscopy; variable strain-rate drive; multiple surface diagnostics	Proton radiography; high-energy transient x-ray diffraction	Next generation temporal control for x-rays; optical ultra-fast lasers	Limits on duty cycle; phase contrast imaging	High-intensity ultra-fast laser system

Another way to state functional requirements:

- **X-rays**
  - That can penetrate the right size samples (<1 mm)
  - For diffraction measurements
  - With the brilliance needed for the right time and spatial resolution
  - And with coherence to allow CXDI
- **Radiography**
  - With the right spatial AND temporal resolution AND field-of-view
  - With appropriate penetration
- **Lasers**
  - For optical pump-probe with ultrashort (atomic) time scales
  - To dynamically drive stress at variable strain
  - With high intensity to create other required probes
- **Available in environmental extremes (radiation, dynamics, temperature), …**
- **With as many other options as cost-benefit-risk allows**

D: Technical Alternatives

High-energy coherent x-ray imaging; dynamic electron microscopy; variable strain-rate drive; multiple surface diagnostics	Proton radiography; high-energy transient x-ray diffraction	Next generation temporal control for x-rays; optical ultra-fast lasers	Limits on duty cycle; phase contrast imaging	High-intensity ultra-fast laser system
FLASH **LCLS** **SCSS** **XFEL**	**GSI; FAIR** **BNL** **J-PARC**	**APS** **Spring-8** **NSLS II**		**Lab-scale systems** **ELI**
Higher energy XFEL; Fast rep rate, but Low duty cycle; multiple probes	Electron radiography for resolution, proton for penetration			Pre-bunched for temporal control; swept energy; high-intensity laser system

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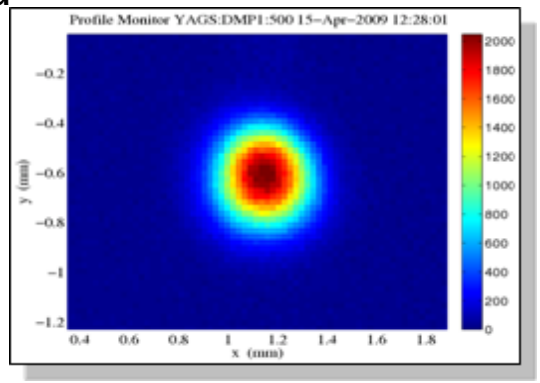
Technical Alternatives: Hard X-ray FELs

- **Free-electron lasers provide high brilliance and coherent x-rays.**
- **Present systems use SASE (self-amplified spontaneous emission) that results in highly irregular temporal structure of micro-pulse.**
- **LCLS (Stanford) and SCSS (Japan) currently plan to operate <8.4 keV (first harmonic); European XFEL (DESY) will be <12keV. (Presently limited by SASE quantum fluctuations and emittance of linac beam.)**
- **Repetition rate of micro-pulses and duty cycle differ:**
  - LCLS <120 Hz
  - SCSS ?
  - XFEL uses superconducting linac and will operate at 5 MHz

8-12 keV XFELs are the transformative tools of *right now!*

European XFEL @ DESY has broken ground (first light ~2015)

LCLS @ SLAC lased on first try (4/10/09), exceeded expectations!



Technical Alternatives: Protons

- **Proton rings and linacs have and can be used for radiography:**
  - Proton radiography invented at Los Alamos and used for dynamic and SNM experiments at 0.8 GeV.
  - AGS at Brookhaven has demonstrated 24-GeV radiography; however, BNL will not allow dynamic classified experiments.
  - GSI at Darmstadt is planning [in collaboration with LANL] 4.5-GeV 4-pulse capability (PRIOR) as prototype for Plasma Physics hutch at FAIR.
- **Storage rings have limited temporal coverage, but can get more charge per micro-pulse.**
- **Linacs can provide more temporal coverage, but may need multiple micro-pulses to get enough charge to do a low-noise radiograph.**

Technical Alternatives: 3-GL Light Sources

- **Synchrotron light sources have excellent access and temporal coverage. They also are most flexible in terms of energy coverage, and can provide very-hard (>50 keV) x-rays. However, brilliance may not be sufficient to make measurements on the shortest transient time scales.**
- **They can provide coherent light, but at even more reduced brilliance making them ineffective for transient measurements.**
- **The DC-CAT (Dynamic Compression Collaborative Access Team) effort at APS at Argonne should help define the ability of 3-GL sources to meet these functional requirements.**

Technical Alternatives: Ultrafast and High-Irradiance Lasers

- **Advances in nano-second laser technology in the 1980's and 1990's has led to NIF.**
- **Advances in picosecond laser technology in the 1990's and 2000's has led to the current generation of "Petawatt" lasers:**
  - OMEGA-EP at Rochester; Texas Petawatt at Austin; Z-Petawatt at Sandia; Jupiter facility at Livermore
  - These lasers all address science of relativistic electrons above 10^{18} W/cm^2.
- **Advances in femtosecond laser technology now will open the "high intensity" frontier ~10^{24} W/cm^2 where ion quiver motion becomes relativistic, synchrotron damping affects the electrons, and there may be vacuum polarization effects**
  - Important for pulsed ion and neutron sources
  - Extreme Light Infrastructure in Europe is example of future facility

Table 1 of Current Facility options against function requirements

		Hard XFELs				Proton Facilities			3GL sources			ultrafast lasers		
	LCLS	SCSS	XFEL	LANSCE	BNL	J-Parc	SNS	GSI/FAIR	APS	NSLS II	LUMOS	Vulcan	ELI	NIF
X-rays														
Max. energy 50-100 keV														
Min. energy <5-10 keV														
Coherency														
Brilliance														
Gating time of single measurement < 1 psec														
Minimum time resolution between two measurements < 350 psec														
Appropriate temporal coverage (# measurements in macropulse, macropulse rep-rate)														
Appropriate micropulse quality (reproducibility, time-smooth, synchronized, ...)														
Bandwith (TBD)														
Sweep frequency >2% in macropulse														
Charged Particle Radiography														
Spatial resolution <100 nm														
Temporal resolution <1ns														
# of measurements in macropulse >10														
Spatial resolution <20 micron														
Temporal resolution <100 microsecs														
Density accuracy <1%														
Optical Laser														
<30 fsec pump laser														
>1kJ drive laser												2.6		1800
high-irradiance system >10^23 W/cm^2														
Dynamic Extremes														
Measure transient (<10 ns) dynamic material temperature to 5%														
High Explosives														
Classified Experiments														
A/B for <11 PEG														
Pressure to 2Mbar														
Strain rate <1e-3 and >1e-6														
Magnetic field steady-state < 20T														
Thermostat 1-few thousand Kelvin @ 5% accuracy and control														

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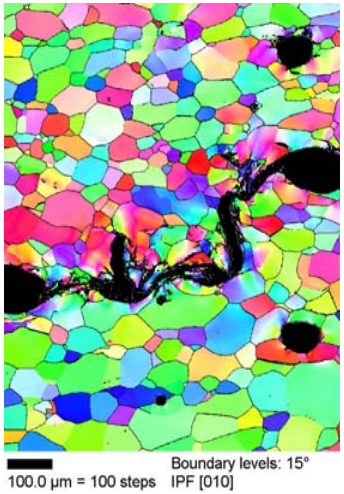
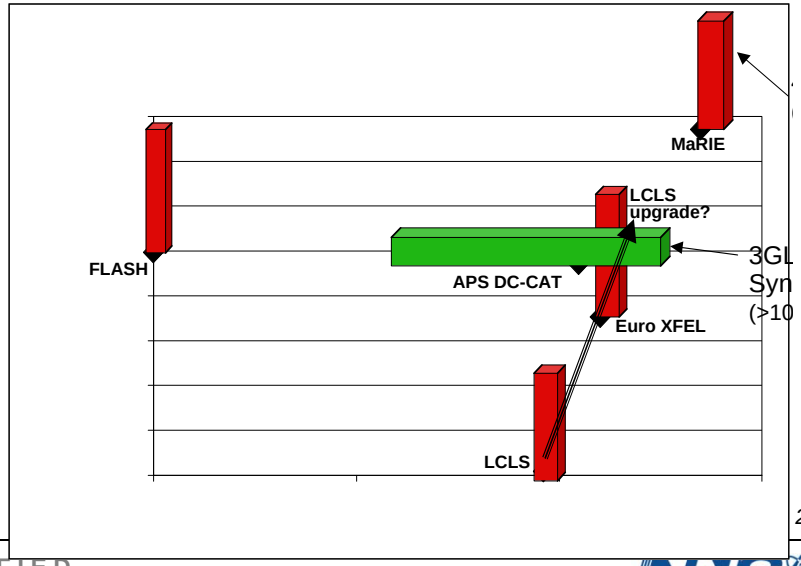
Through Multi-Probe Diagnostic Hall, MaRIE provides unique scattering and imaging capabilities to bridge the micron gap in extreme environments

- **High-energy (50-115 keV) photon source (for multigranular sample penetration) with high intensity (to resolve transient effects) and coherency and high repetition rate (quantitative imaging of dynamic processes)**
  - Preferred alternative is 4GL XFEL light source (low duty cycle to reduce cost)
  - Can provide 3-dimensional dynamic structure information
- **Charged particle microscopy to provide simultaneous measurements to constrain information at many scales**
  - Preferred alternative is at 0.8 GeV, with higher current (better time resolution & FFMF drivers) and better optics (for better spatial resolution)
  - Examining alternatives for protons and electrons
- **Flexibility in creating material environments (pressure, strain, temperature, …)**
  - Robust suite of dynamic loading and material heating techniques
- **Couple probes with *in-situ* irradiation and controlled synthesis**
  - ultra-fast/ultra-short *in-situ* microscopies
  - initial synthesis and post-mortem characterization





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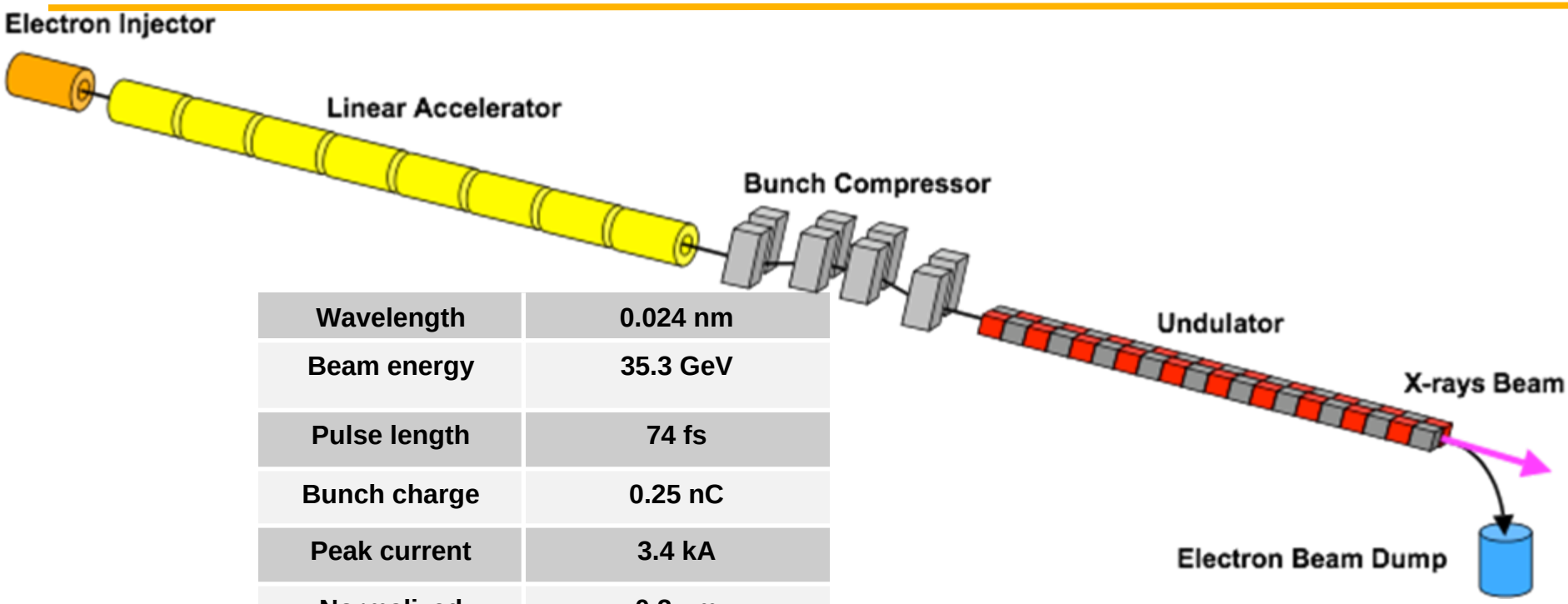
Recent Technical Analysis Advancing the Alternatives

- **XFEL**
  - Flat-Beam-Transform and Emittance Exchange techniques allow reducing transverse emittance to beat quantum fluctuation limit and allow lower energy electrons to make 50-keV photons (20-GeV linac). Allows shorter undulators, lower cost, and better meet functional requirements.
  - Exploring methods for better temporal control (non-SASE)
  - Working on sweeping energy in macropulse
  - Analyzing trade-offs on duty cycle, temporal coverage, and cost

- **Electron radiography**
  - For <1 mm samples, get <100 nm spatial resolution at 20 GeV
  - EGS5 calculation of few degrees K heating per image
  - Laser photocathode has demonstraated 27 nC bunches => great temporal resolution, one radiography per micropulse.

MaRIE 50-keV XFEL Original Preferred Concept



Wavelength	0.024 nm
Beam energy	35.3 GeV
Pulse length	74 fs
Bunch charge	0.25 nC
Peak current	3.4 kA
Normalized rms emittance	0.2 μm
rms Energy spread	0.01%
Undulator period	3 cm
Undulator K_{rms}	2.624
Undulator type Length	Planar hybrid PM 20 m

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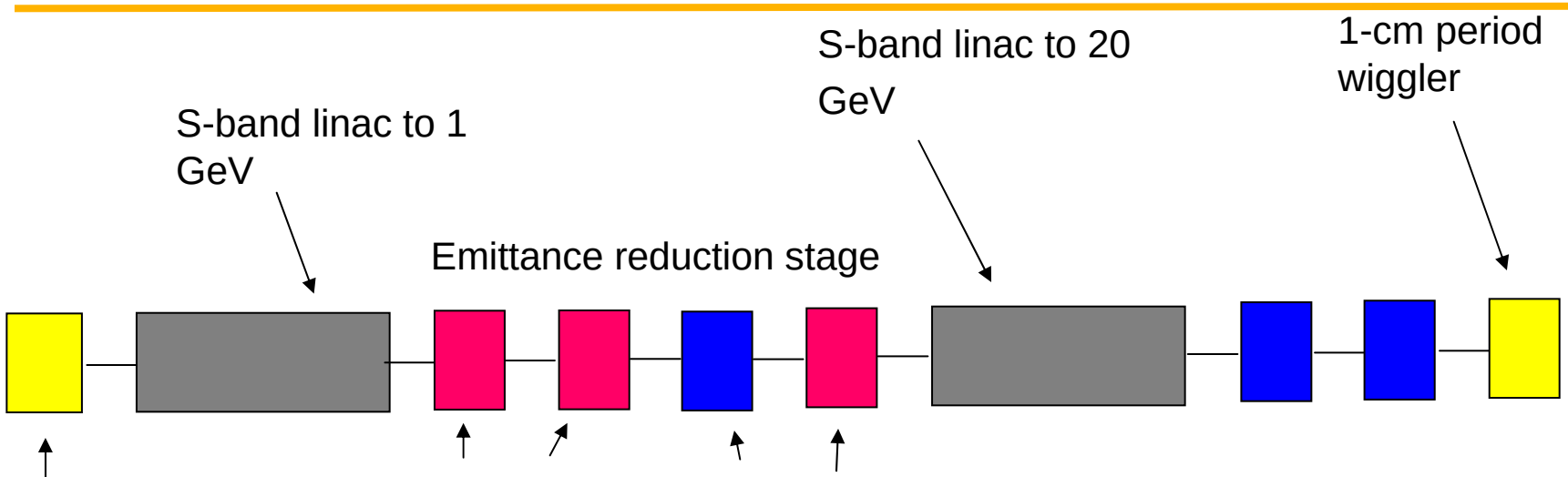
What We Are Thinking For Preferred Alternative



500 kV DC injector
or AFEL copy
or CTF3 copy
0.50 nC, L=35 μm
$\varepsilon_x \sim 0.7$ μm
$\varepsilon_y \sim 0.7$ μm
$\varepsilon_z \sim 1.4$ μm

FBT 1: L=0 μm
$\varepsilon_x \sim 70$ μm
$\varepsilon_y \sim 0.007$ μm
$\varepsilon_z \sim 1.4$ μm

FBT 2: L=2 μm
$\varepsilon_x \sim 35$ μm
$\varepsilon_y \sim 0.014$ μm
$\varepsilon_z \sim 1.4$ μm

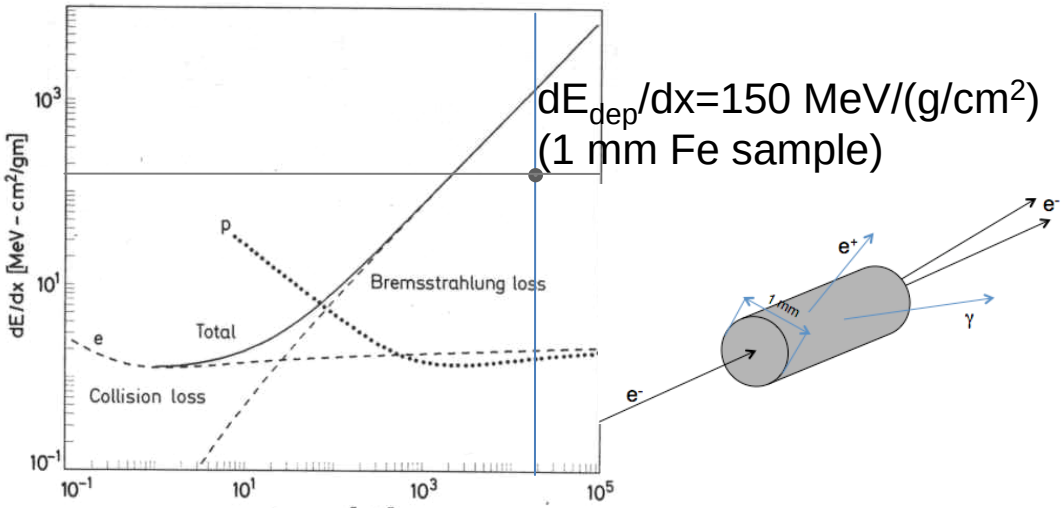
EE 1: L=2 μm
$\varepsilon_x \sim 1.4$ μm
$\varepsilon_y \sim 0.014$ μm
$\varepsilon_z \sim 35$ μm

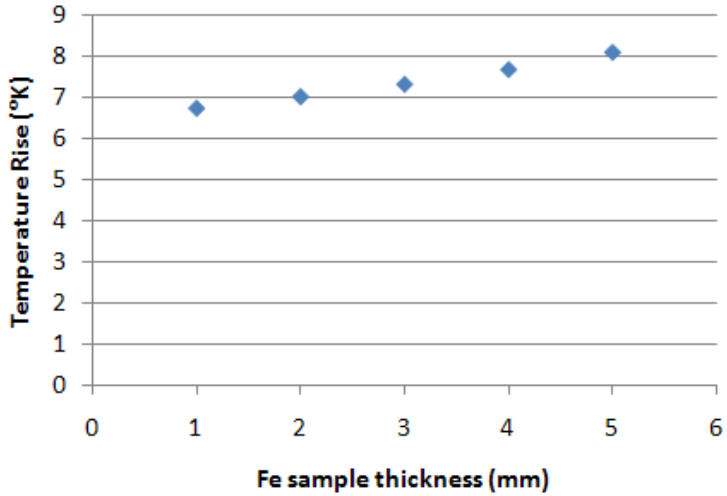
FBT 3: L=0 μm
$\varepsilon_x \sim 0.14$ μm
$\varepsilon_y \sim 0.14$ μm
$\varepsilon_z \sim 35$ μm

EE 2: L=0 μm
$\varepsilon_x \sim 100$ μm
$\varepsilon_y \sim 0.14$ μm
$\varepsilon_z \sim 0.14$ μm

EE 3: L=0 μm
$\varepsilon_x \sim 0.14$ μm
$\varepsilon_y \sim 0.14$ μm
$\varepsilon_z \sim 100$ μm

20-GeV Electron Radiography can be used for <1 mm samples as a high-resolution probe of microstructure dynamics





- **Electron limitations are the same as protons. New scattering and energy loss processes are introduced because of the light mass of the electron.**
- **For thin samples, there are enough non-Bremsstrahlung interacting, forward electrons to make useful radiographs.**
- **EGS5, developed by KEK, has recently included electron scattering through Bremsstrahlung radiation. Heating per radiograph acceptable.**

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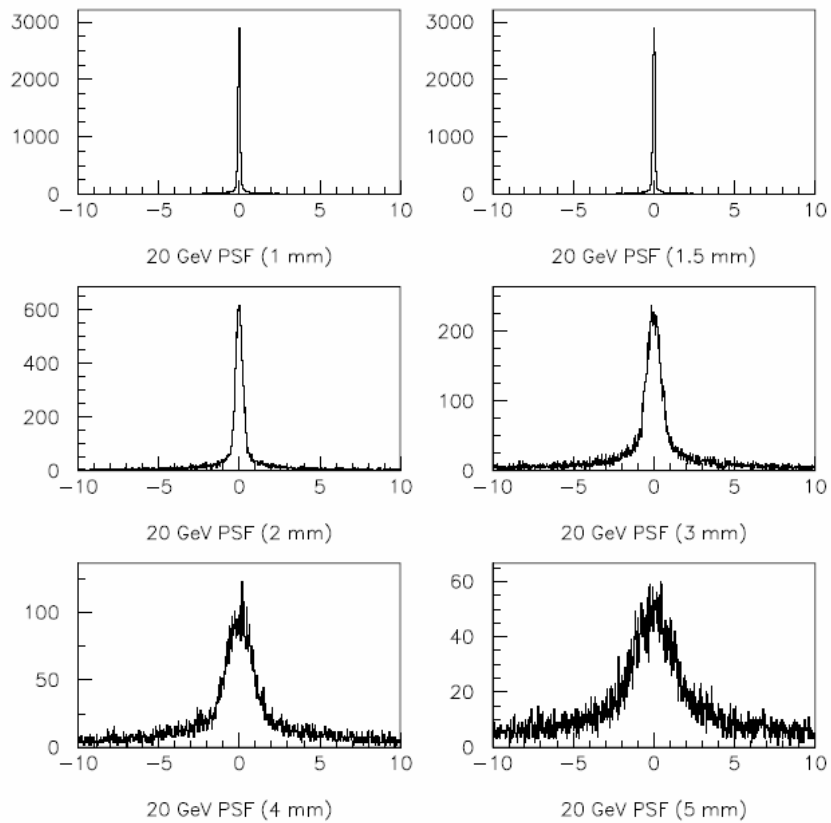
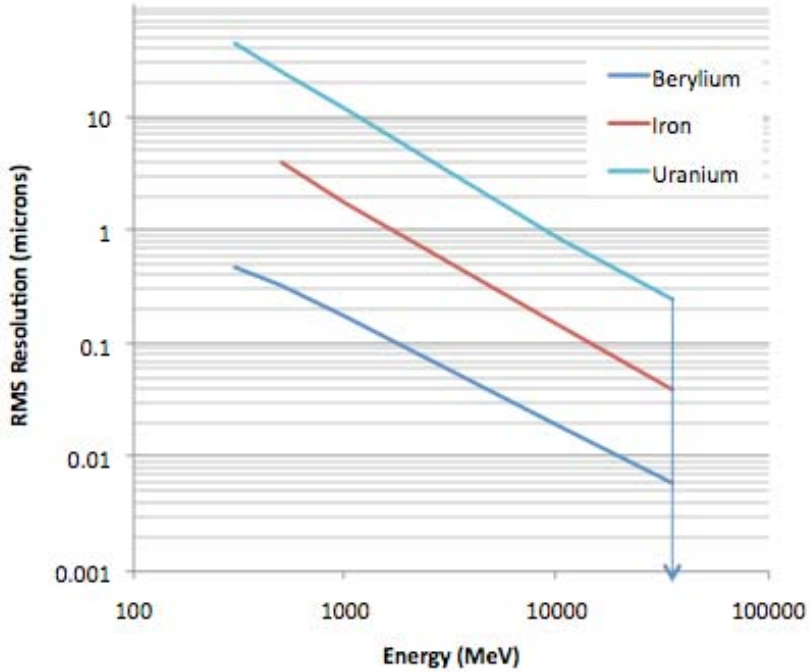
20-GeV Electrons are an Ideal Probe for <1mm objects



Iron Object of Various Thicknesses



Considering design of a magnifier for 14-GeV electrons at LCLS.

Photo-injector technology allows variations in charge per micro-pulse between 0.5 nC and 27 nC.

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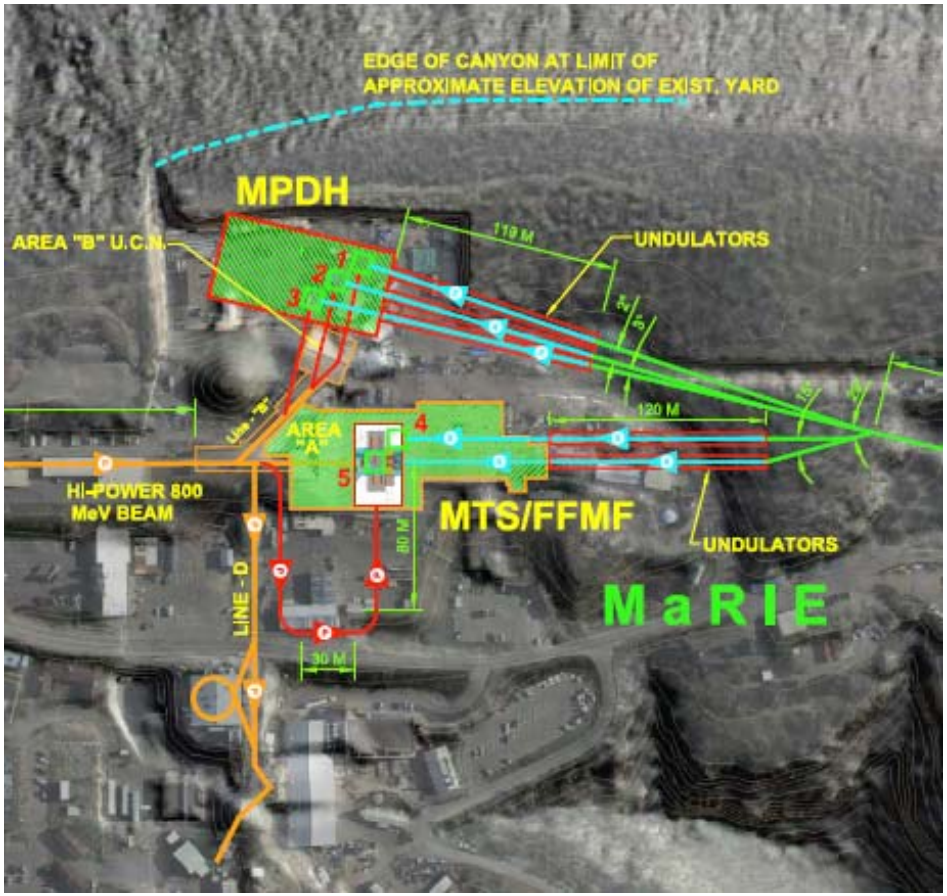
Describe the MaRIE Preferred Technical Alternative

- **20-GeV linac using advanced emitance control to provide 50-150 keV brilliant coherent light with high repetition rate and low duty cycle.**
  - Exploring techniques for advanced temporal control
- **Use both electron and proton linacs for radiography**
- **Laser hall with ultra-fast, drive, and high-intensity systems**
  - Hall
    - Hutch
      - End-Station
        - Diagnostic/Instrument



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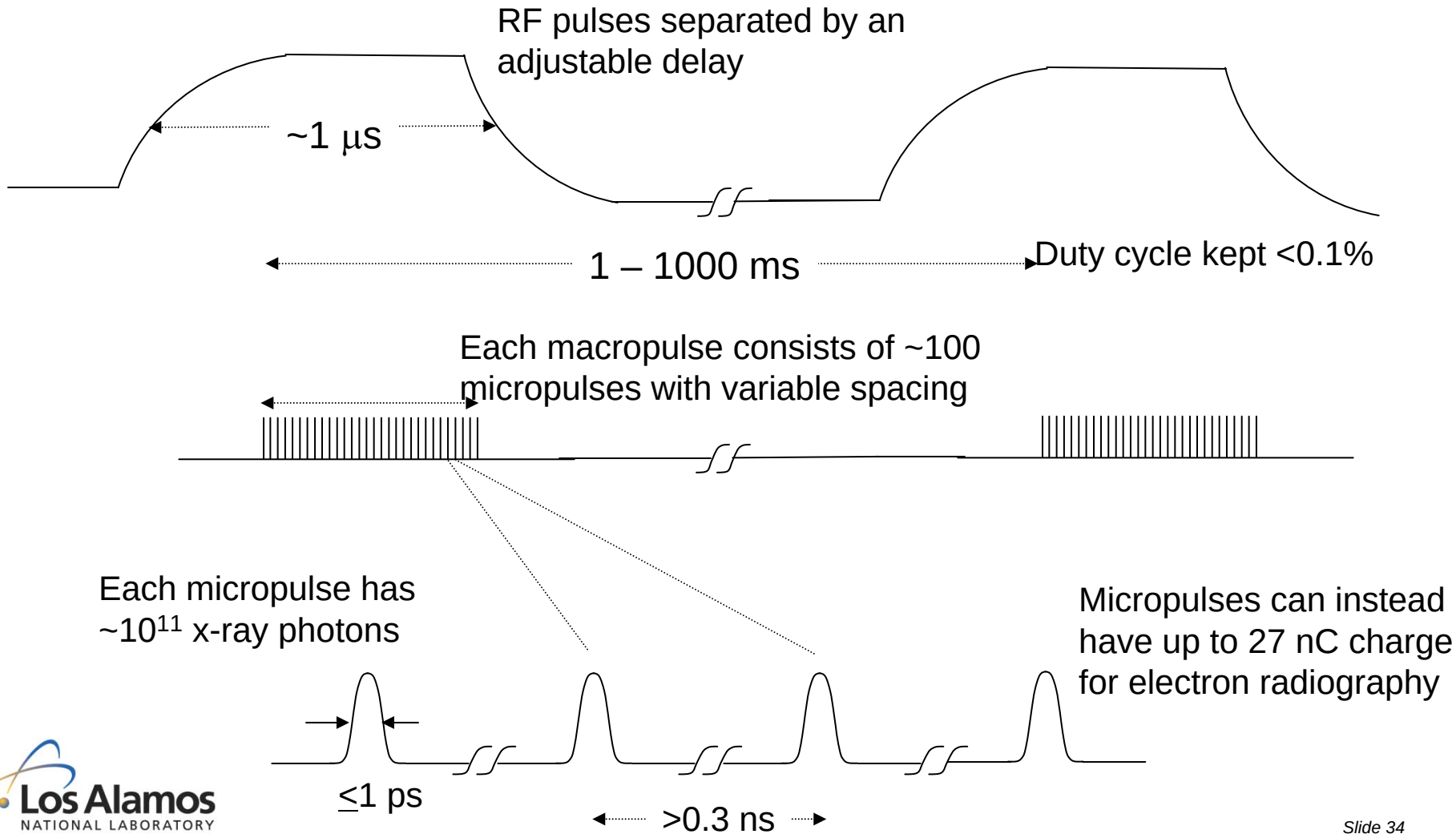
MaRIE XFEL temporal pulse format provides high repetition rate at affordably low duty cycle



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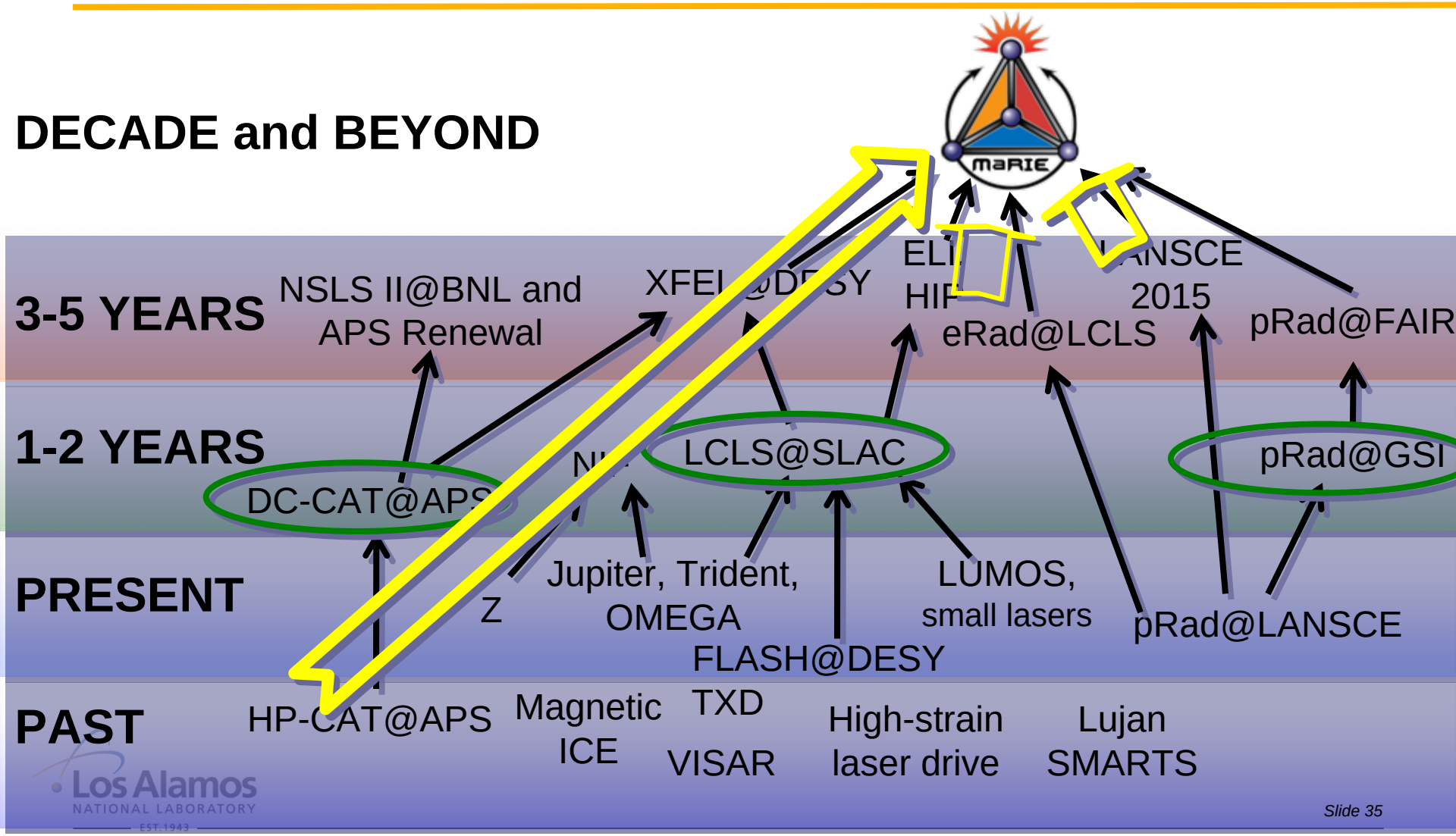
The Roadmap from DC-CAT to MaRIE



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What can you do to contribute to MaRIE?

Define frontier experiments that are killer apps

Help quantify the requirements for future science

Analyze the technical alternatives

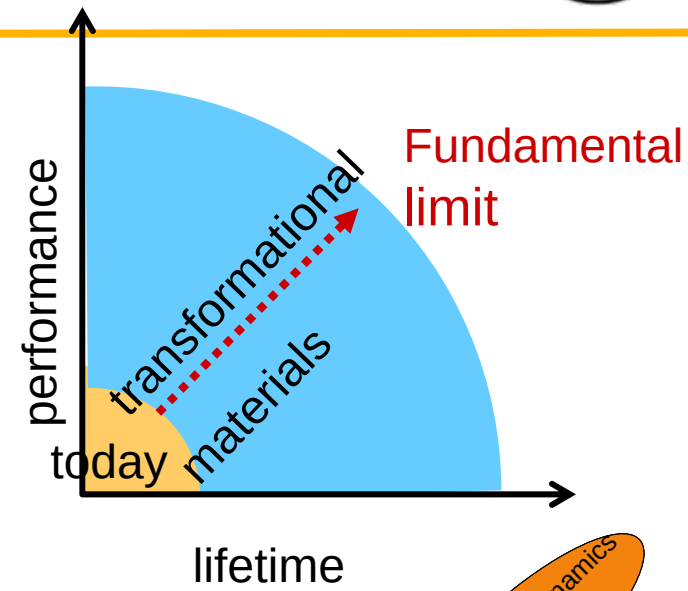
Propose preferred alternatives

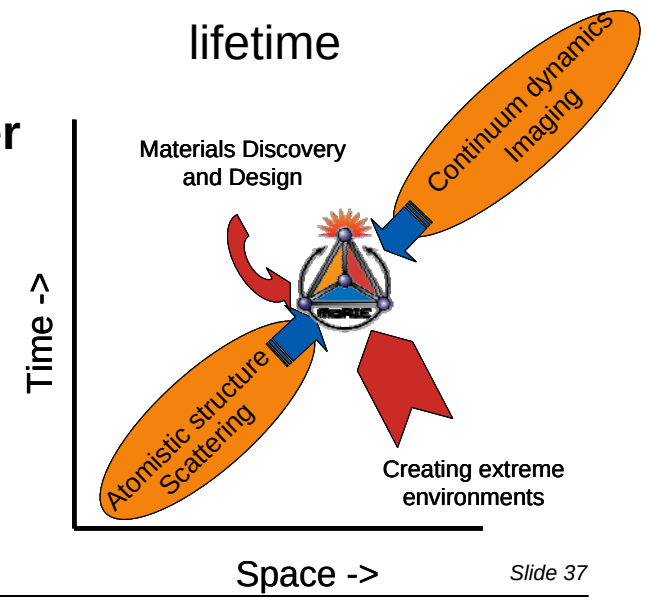
Help us Document the case for MaRIE!

The transition from "observation & validation" to "prediction & control" is a central mission challenge AND the frontier of materials research

- **Conquering "the micron frontier" is essential for solving transformational materials grand challenges**
- **MaRIE will provide unique capabilities**
  - Simultaneous *in situ* imaging and scattering measurements
  - Accessing materials irradiation/damage extremes
  - Incubating materials discovery and solutions through control of defects and interfaces
- **MaRIE provides unprecedented international user resources in a national security science setting**
- **LANSCE is essential for MaRIE's success**

MaRIE will be the first capability with unique co-located tools necessary to realize transformational advances in materials performance in extremes





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