FAST X-RAY IMAGING AND DIFFRACTION

for Engineering Materials Science and Mechanics

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26th National School on Neutron and X-Ray Scattering, Argonne, July 30, 2024

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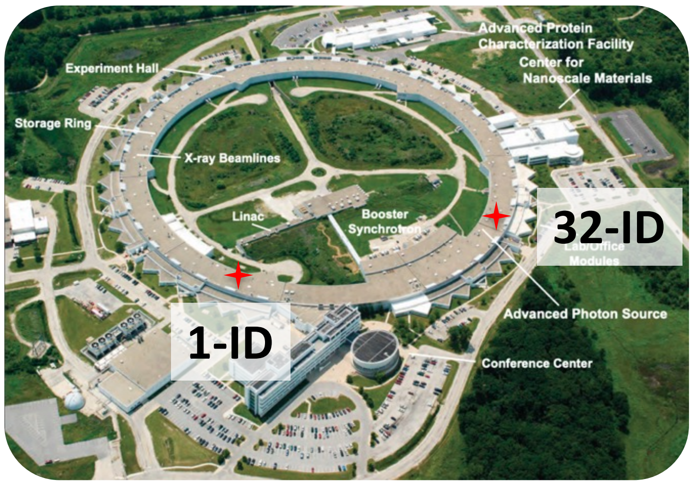


Imaging: Dr. Kamel Fezzaa and Dr. Samuel Clark
Diffraction: Dr. Andrew Chuang

I. **High-speed x-ray imaging at the Advanced Photon Source**

II. **Fast diffraction experiments at different time scales**

III. **Operando synchrotron experiments on metal additive manufacturing**

QUESTIONS WE WILL ANSWER TODAY

1) **What is special about engineering materials science?**

2) **What are the main advantages of synchrotron over lab-source?**

3) **What make APS a unique facility for high-speed x-ray experiments?**

4) **What affect the spatial and temporal resolutions of fast imaging and diffraction?**

"FAST" PROCESSES IN ENGINEERING SCIENCE

Real materials under real conditions in real time

- Millimeter sample size to represent bulk behavior
- Complex system to deliver realistic work conditions

- Fluid dynamics
- Energetic materials and rapid reactions
- Dynamic loading
- Materials machining and processing
- Additive manufacturing

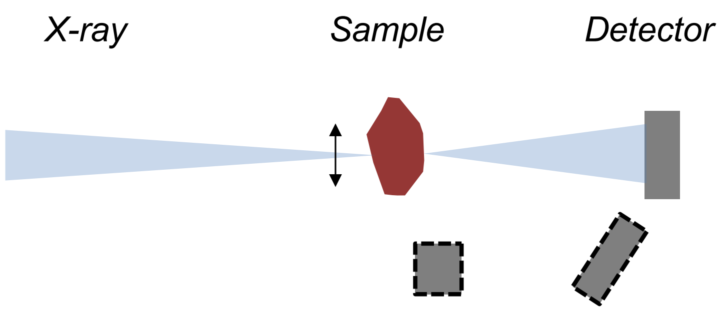
Dynamic irreversible and non-repeatable materials and engineering processes





X-RAY IMAGING AND MICROSCOPY TECHNIQUES

❑ **Scanning probe microscopy**



- Fluorescence contrast
- Absorption contrast
- Absorption fine structure contrast
- Scattering contrast
- Diffraction contrast
- Computed tomography (3D)

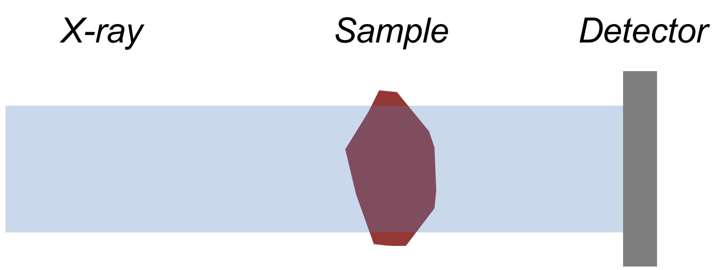
Spatial resolution: probe size

❑ **Coherent imaging**

- Ptychography
- Coherent diffractive imaging

Spatial resolution: q range

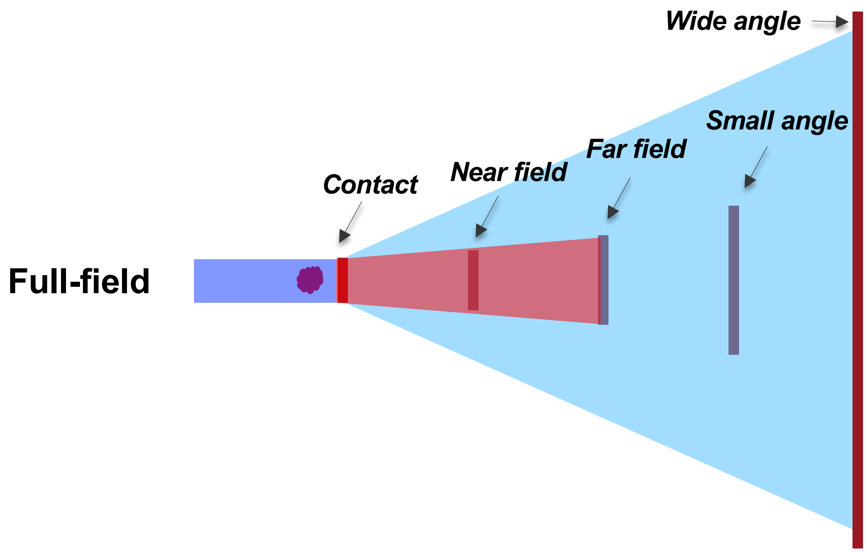
❑ **Propagation-based full-field imaging**



- Absorption contrast
- Phase contrast imaging
- Projection microscopy
- Transmission x-ray microscopy
- Diffraction contrast
- Computed tomography (3D)

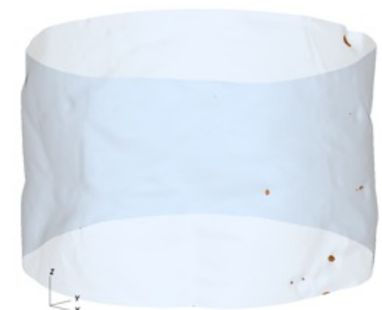
Spatial resolution: detection pixel size

PROPAGATION-BASED FULL-FIELD X-RAY IMAGING

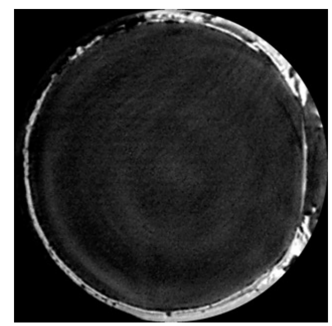


3D

X-ray micro computed tomography (μ-CT)



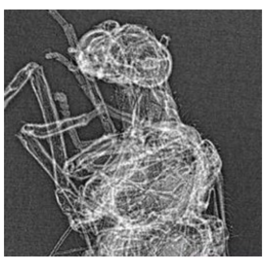
Transmission x-ray microscope (nano CT)



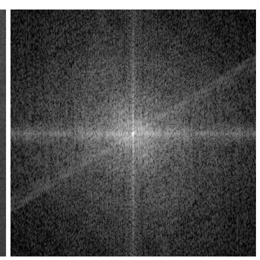
2D

Absorption contrast imaging

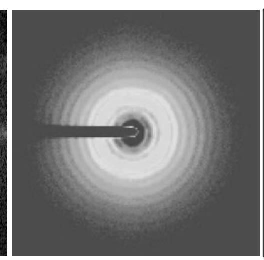
Phase contrast imaging



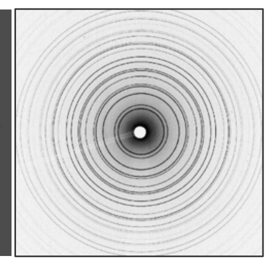
Coherent diffractive imaging



Small-angle scattering



Diffraction or wide-angle scattering



APS 32-ID-B BEAMLINE UNDULATOR SOURCES

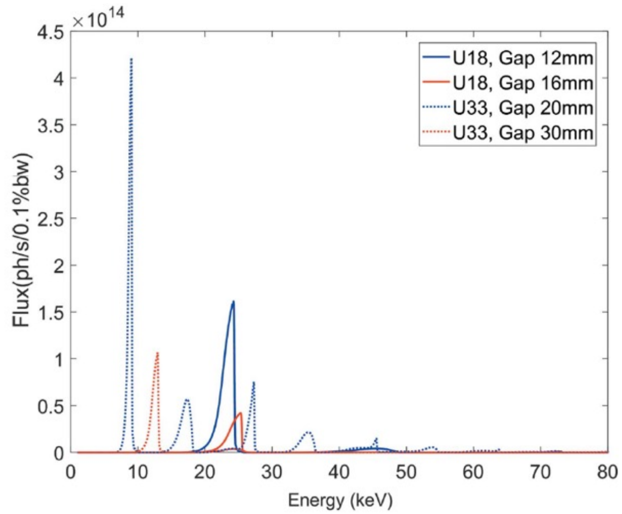
Tandem undulators

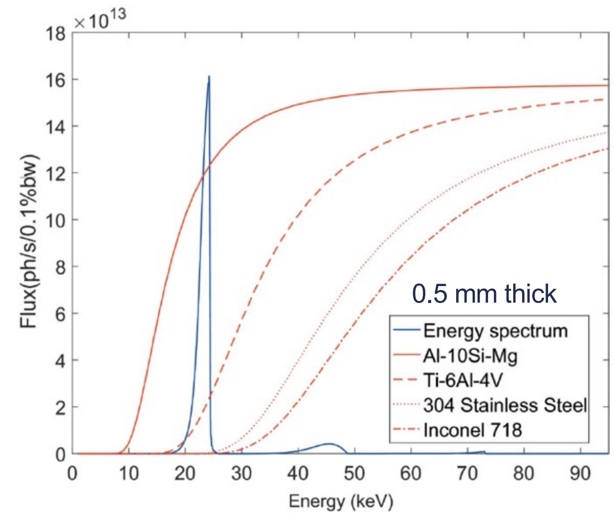
❑ U33 (white beam)

- Length: 2.4 m
- Period: 3.3 cm
- Min Gap: 11 mm
- E1 range: 5~14 keV
- $\Delta E_1/E_1$: 1~2%

❑ U18 ("pink" beam)

- Length: 2.4 m
- Period: 1.8 cm
- Min Gap: 11 mm
- E_1 range: 23.7~25.7 keV
- $\Delta E_1/E_1$: 5~10%

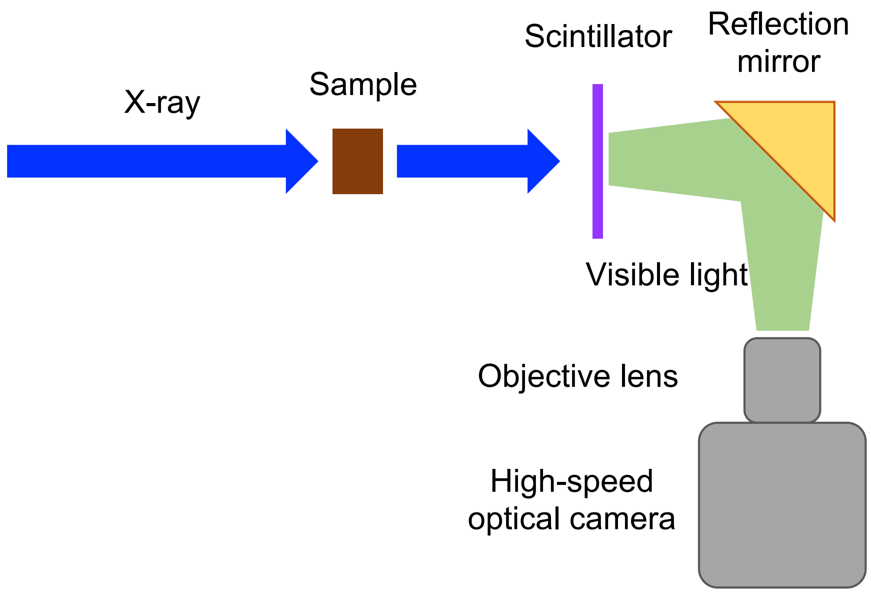


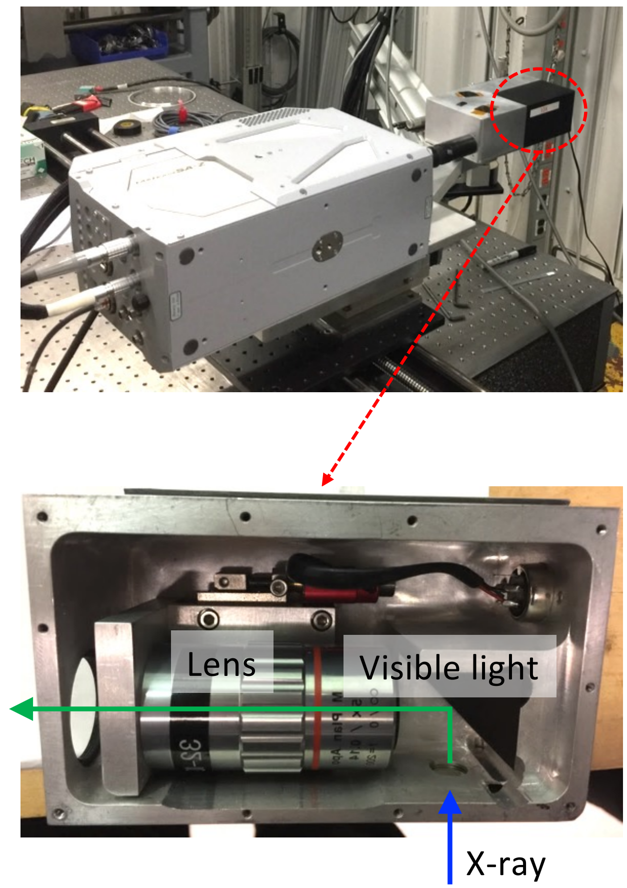


Undulator		Integrated over 1-65 keV		1st harmonic	
Period (cm)	Gap(mm)	Flux*	Singlet	Flux	Singlet
3.3	20	1.8×10^{16}	2.8×10^{9}	1.3×10^{16}	2.0×10^{9} (71%)
	30	4.7×10^{15}	7.3×10^{8}	4.5×10^{15}	6.9×10^{8} (95%)
1.8	11	4.5×10^{16}	6.9×10^{9}	4.1×10^{16}	6.3×10^{9} (92%)

* Unit: ph/s/0.1%BW, 1.5x1.5 mm^2 beam size

HIGH-SPEED X-RAY IMAGING DETECTION SYSTEM





Scintillator-couple optical detection

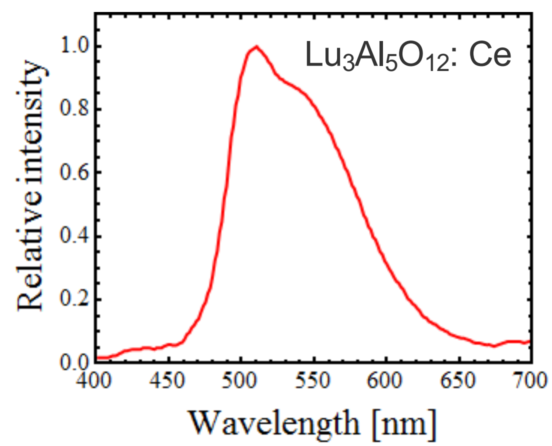
- High spatial resolution: imaging sensor pixel size, magnification by the lens
- High temporal resolution: delay time of scintillator, frame rate and exposure time of camera, x-ray pulse structure

SPATIAL RESOLUTION OF IMAGING

- X-ray beam size: 2 mm x 2 mm
- Camera sensor:
  - CMOS: 18.5 µm/pixel, 1280 x 800, image size reduces as frame rate increases
  - Hybrid CMOS (with on-pixel storage): 30 µm/pixel, 400 x 250, image size remains the same
- Objective lens: 2x, 5x, 10x, 20x
- Scintillator light emission: visible light (wavelength: 400~700 nm)

Phantom TMX-7510

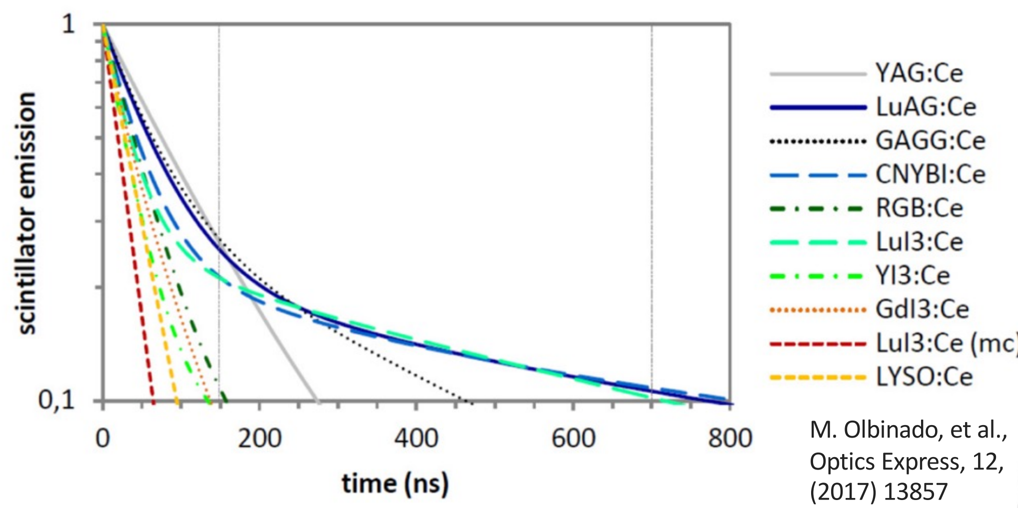
Shimadzu HPV-X2



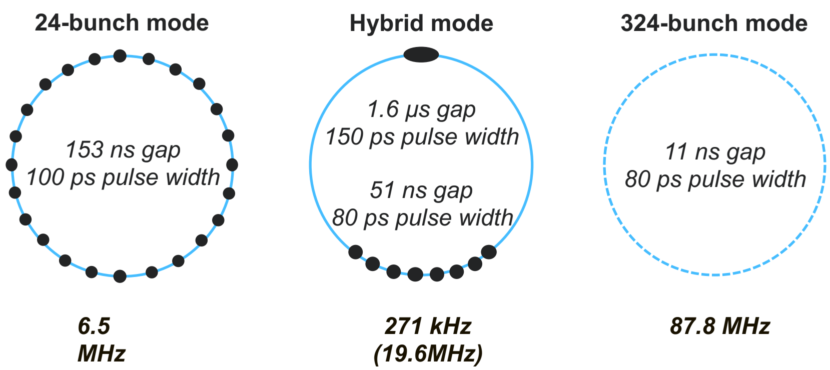
TEMPORAL RESOLUTION OF IMAGING AT APS

- ❑ **Exposure time**:
  - Camera specs (CMOS: 100 ns; Hybrid-CMOS: 50 ns)
  - Scintillator decay time

- ❑ **Frame rate**:
  - Camera specs (CMOS: 1.75 MHz; Hybrid-CMOS: 10 MHz)
  - Needed field-of-view for experiment
  - X-ray pulse structures



M. Olbinado, et al., Optics Express, 12, (2017) 13857

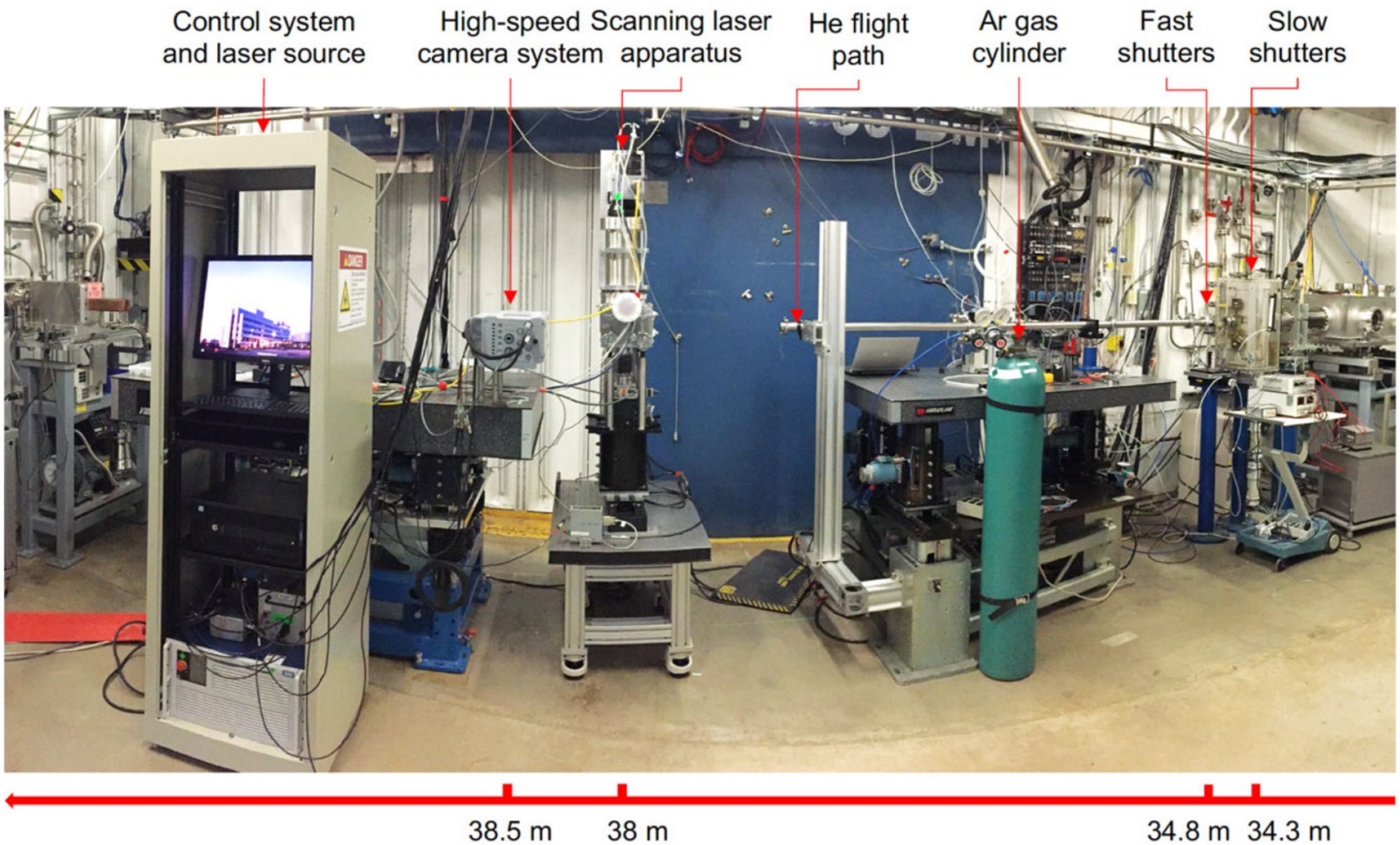


24-bunch mode: MHz imaging with single pulse exposure

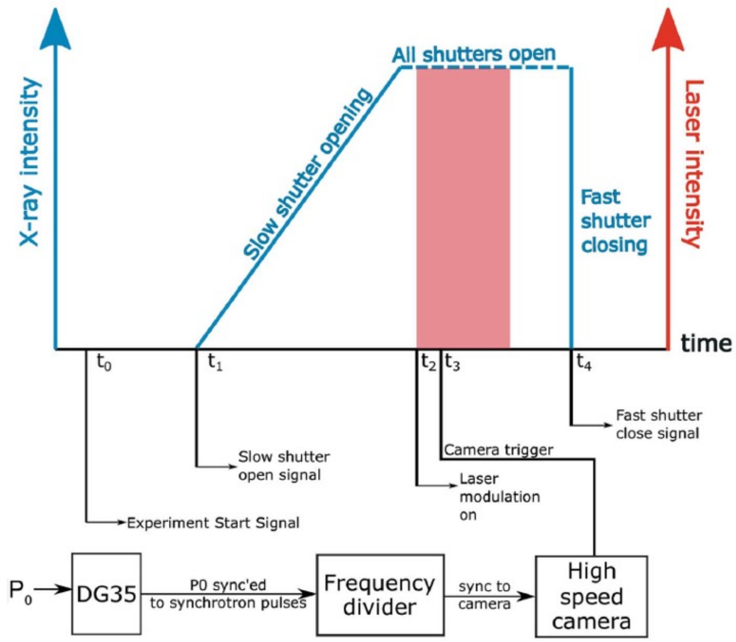
Hybrid mode: Fixed frame rates, but stronger single pulse

324-bunch mode: Experiments with > μs exposure, no intensity fluctuation in each image

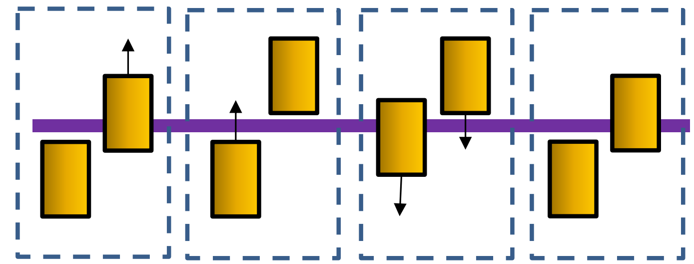
APS 32-ID-B HIGH-SPEED EXPERIMENTAL HUTCH



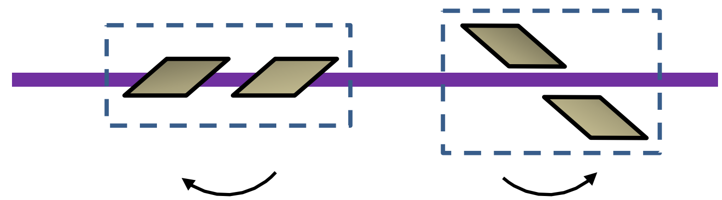
TIMING SCHEME AND CONTROL OF EXPERIMENTS



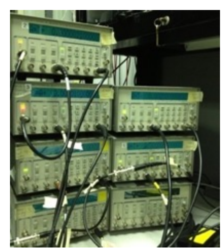
Slow shutters



Fast shutters



Delay generators

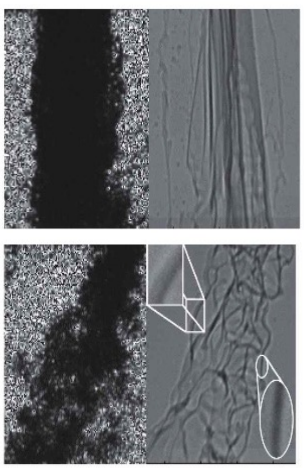


SRS535

SRS645

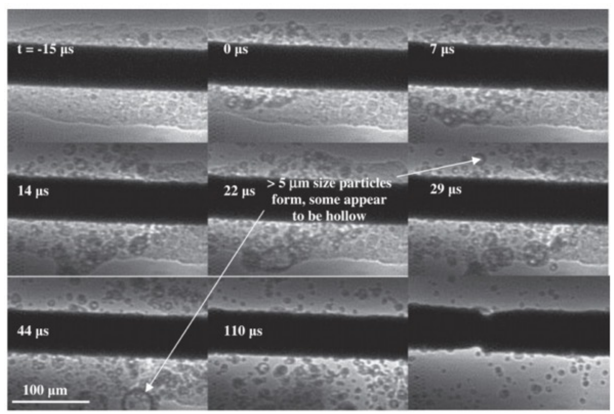
HIGH-SPEED X-RAY TECHNIQUES OF HIGHLY DYNAMIC PROCESSES

Fuel spray: visible light vs X-ray



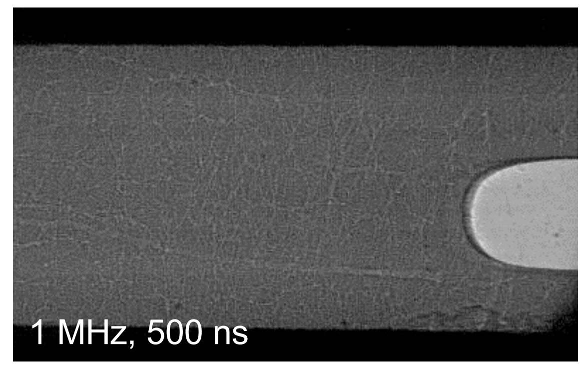
Wang Y. et al. Nature Physics 4, 305-309 (2008)

Thermite reaction: Al-Fe_2O_3



K. Sullivan et al. Combustion and Flames 159, 2-15 (2012)

Fracture of bone upon impact



From Wayne Chen's group, Purdue University

Search Kamel Fezzaa's publications for various high-speed x-ray imaging experiments

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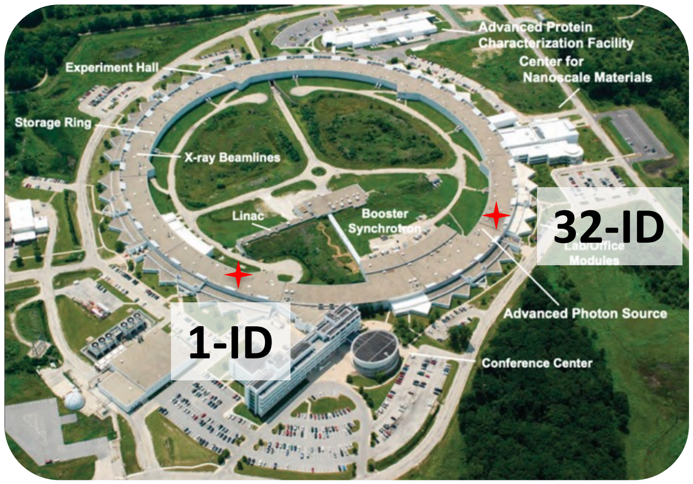

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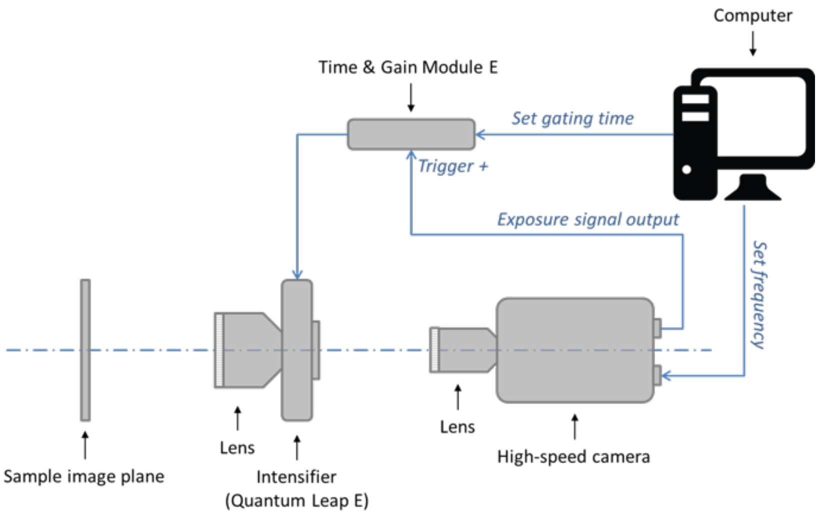
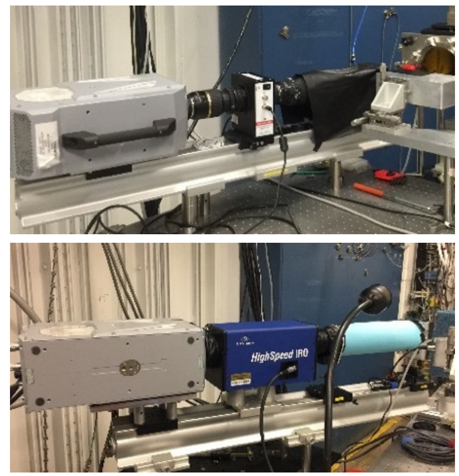
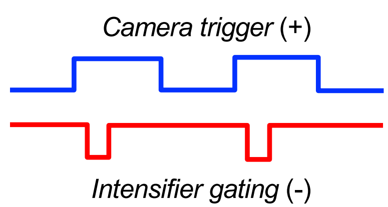
HIGH-SPEED DIFFRACTION DETECTION SYSTEMS AT 32-ID

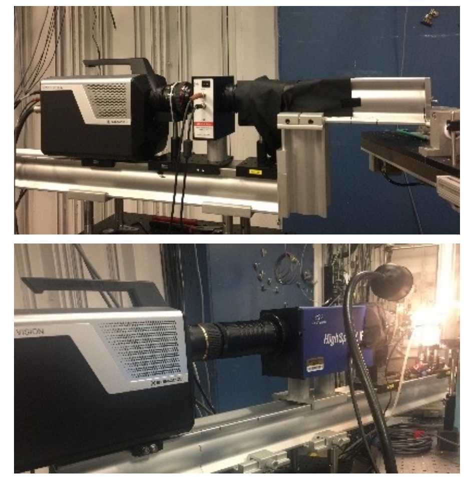


- ❑ Intensifier: LaVision IRO, Quantum Leap
- ❑ Camera: Photron SA-Z, Shimadzu HPV-X2
- ❑ Scintillator: $Lu_{1.8}Y_{0.2}SiO_5$:Ce (LYSO)
  - Thickness: 300 µm
  - Diameter: 65 mm
  - Al front coating

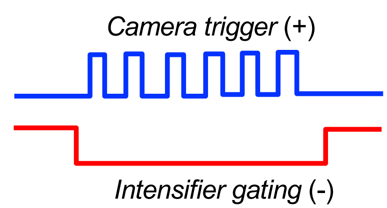


- Camera: Photron SA-Z
- Intensifier trigger: multiple
- Pixels: 1024 x 1024 (60~70 µm/pixel)
- Min exposure: 100 ps
- Max frame rate: 200 kHz
- Fast dynamics spanning 10s' ms



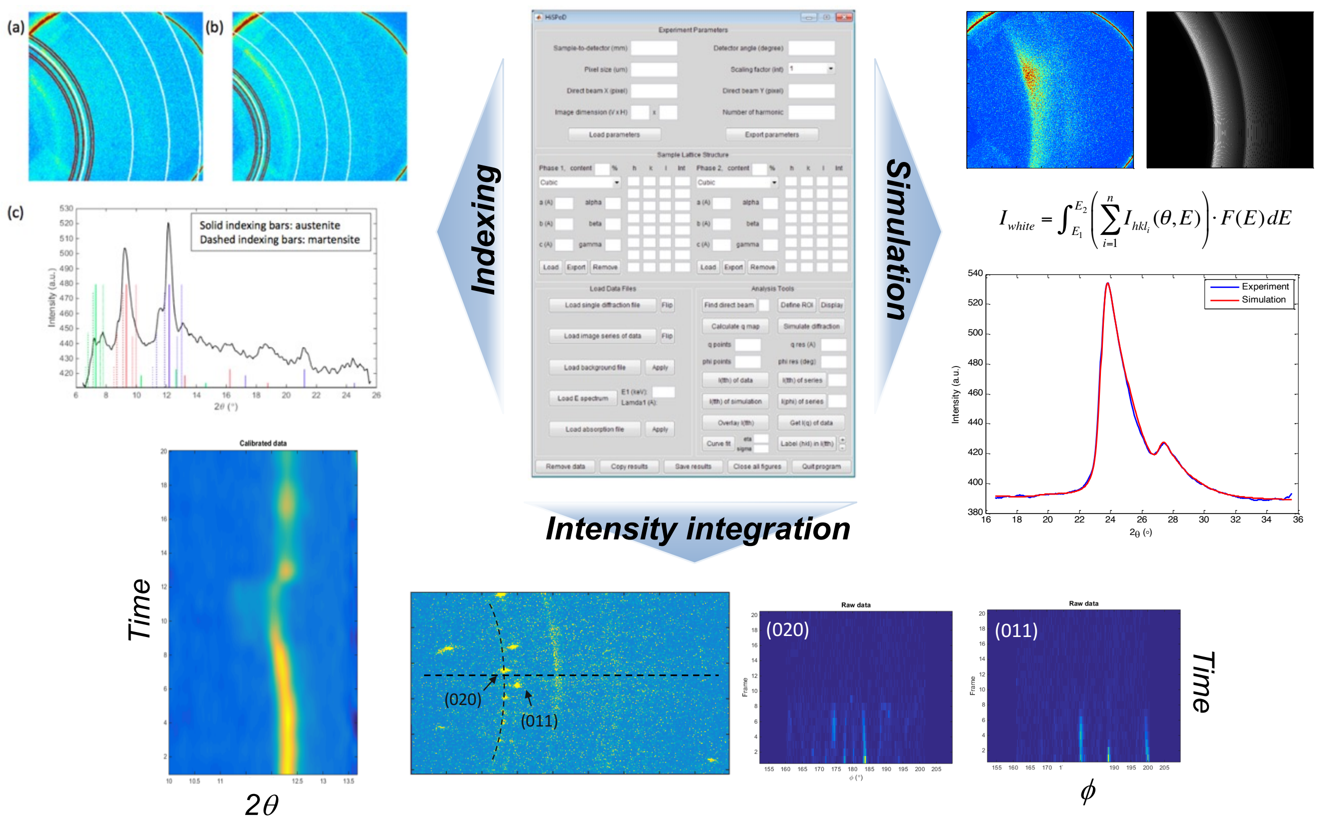


- Camera: Shimadzu HPV-X2
- Intensifier trigger: single
- Pixels: 400 x 250 (60~70 µm/pixel)
- Min exposure: 100 ps
- Max frame rate: 10 MHz
- Ultrafast dynamics spanning 10s' µs



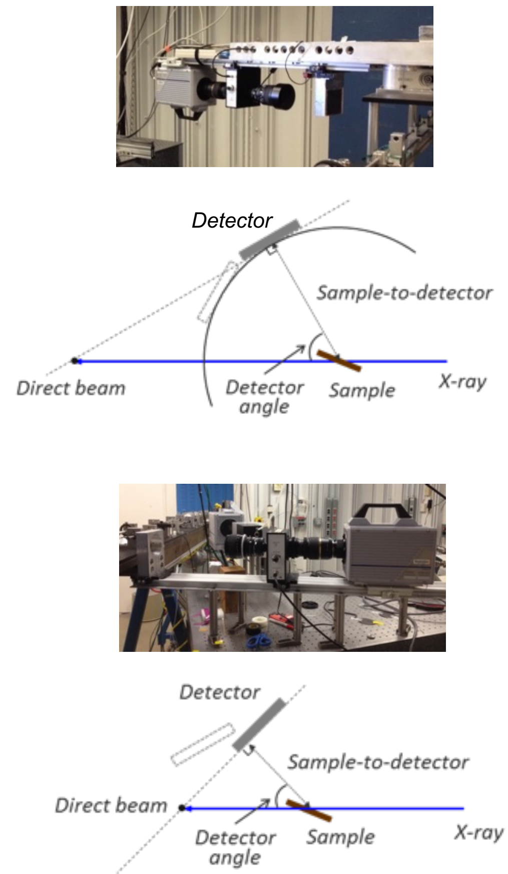
DATA ANALYSIS SOFTWARE FOR WHITE-BEAM DIFFRACTION

HiSPoD: High-Speed Polychromatic Diffraction

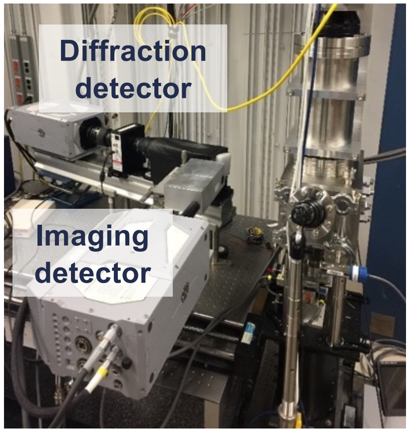


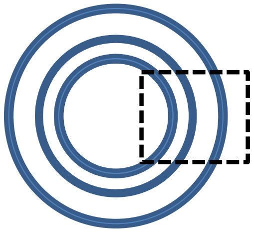
$$I_{white} = \int_{E_1}^{E_2} \left(\sum_{i=1}^{n} I_{hkl_i}(\theta, E) \right) \cdot F(E)\, dE$$

Scattering geometry



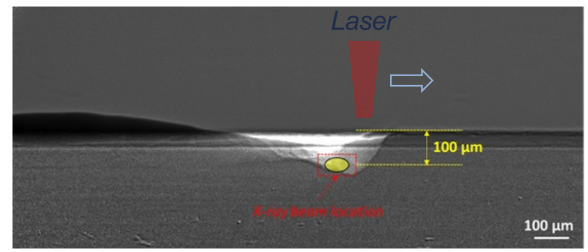
PINK BEAM DIFFRACTION AT 32-ID

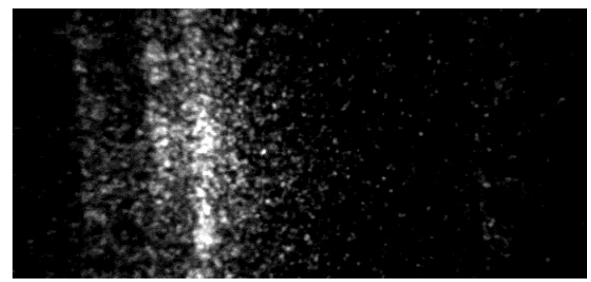




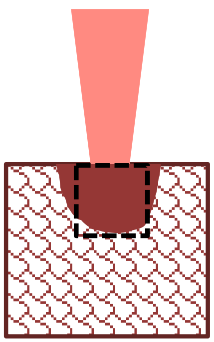
- ❑ **Phase transformation of Ti-6Al-4V**
  α-Ti → melting → β-Ti with coarse grains → α-Ti with fine grains
- ❑ **32-ID source**
  - U18 pink: ~24 keV (1^{st})
  - Bandwidth: ~5%
- ❑ **Detector**
  - Scintillator + intensifier + optical CMOS camera

Scanning laser mode



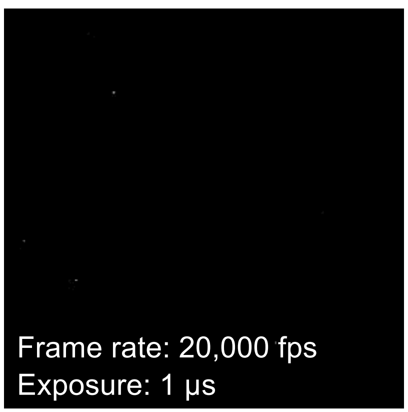


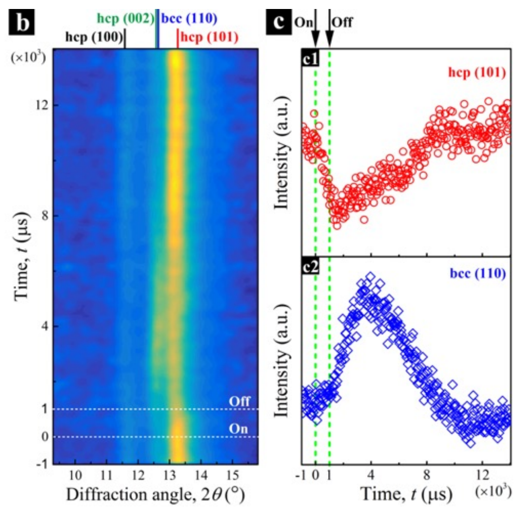
Frame rate: 100,000 fps
Exposure: 5 µs
X-ray beam size: H100 x V60 μm^2



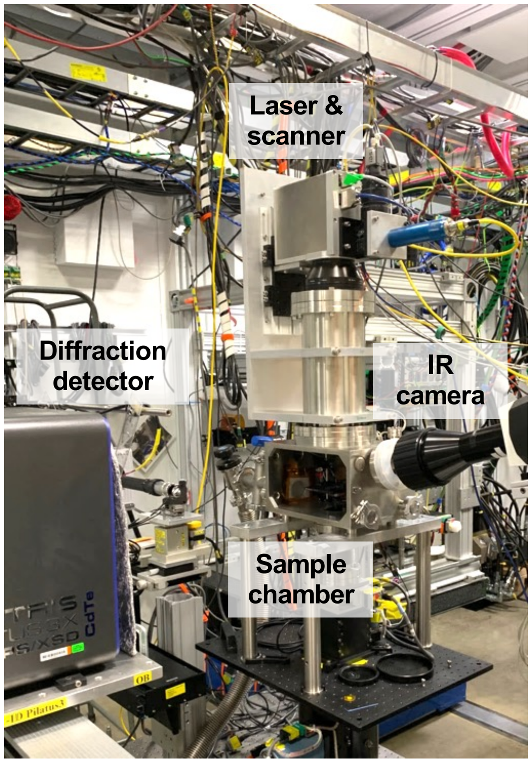
C. Zhao, et al., Scientific Reports, 7, (2017) 3602

Spot welding mode

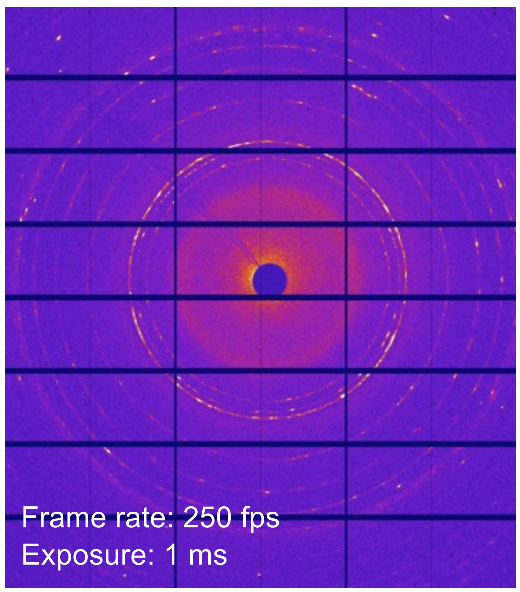


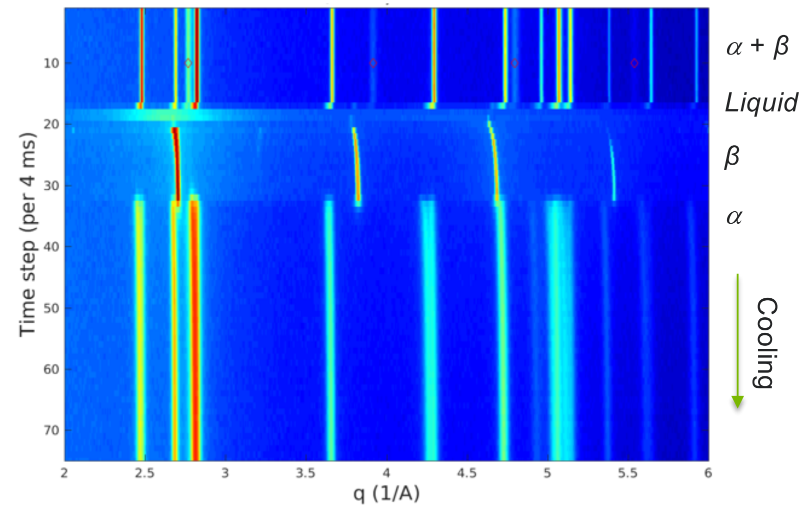


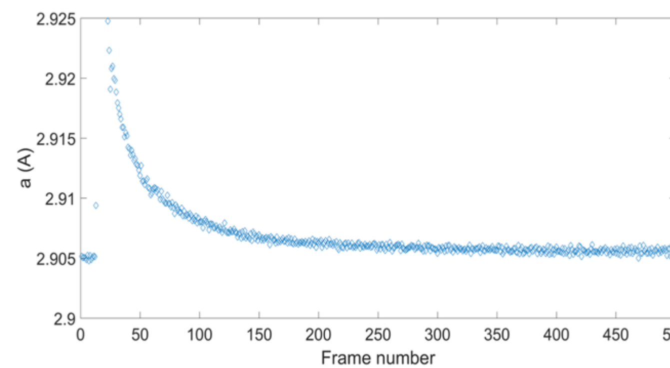
MONO BEAM DIFFRACTION AT 1-ID



- **1-ID source**
  - Superconducting undulator
  - Mono: E = 55.6 keV
- **Detector**
  - PILATUS3X 2M CdTe



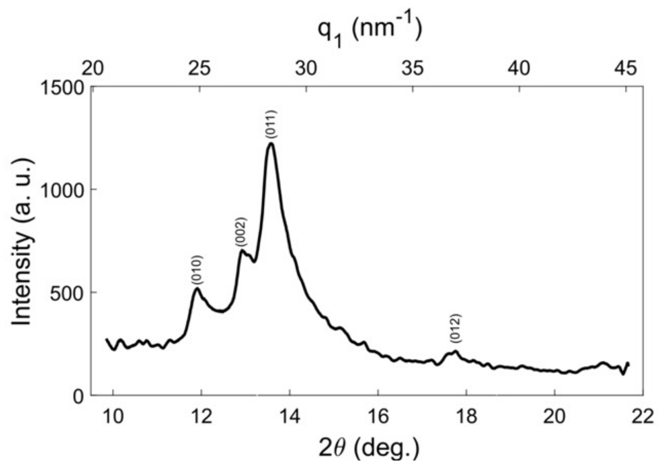




S. Oh, et al., Materials Research Letters, 9, (2021) 429

COMPARISON OF IN SITU DIFFRACTION DATA

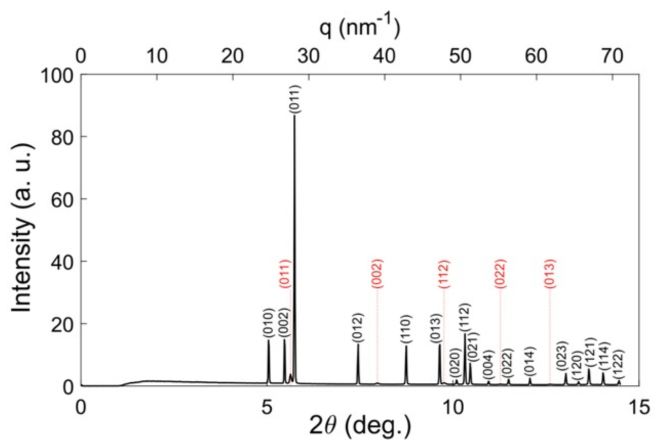
32-ID
pink beam



- X-ray energy: mid-energy pink beam
- Detector: small indirect detection
- Frame rate: 100s' kHz
- Exposure time: microsecond
- Detector dynamic range: low
- S/N: low

Fast, but limited detection

1-ID
mono beam



- X-ray energy: high-energy mono beam
- Detector: large direct detection
- Frame rate: 100s Hz
- Exposure time: millisecond
- Detector dynamic range: high
- S/N: high

Slow, but high resolution

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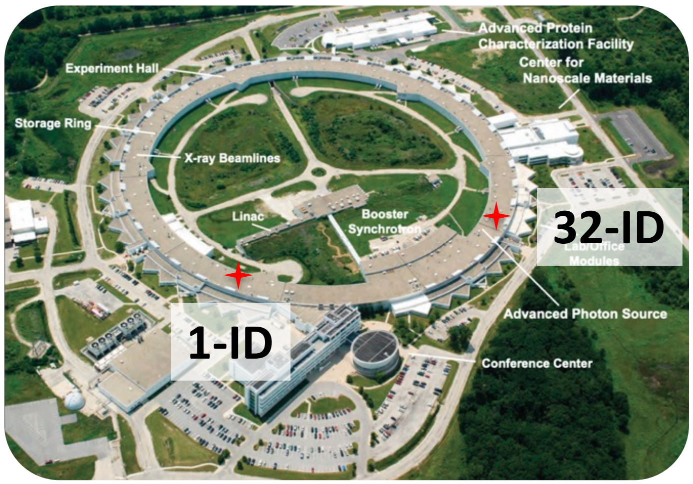

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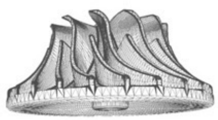
ADDITIVE MANUFACTURING (3D PRINTING)



Computer model

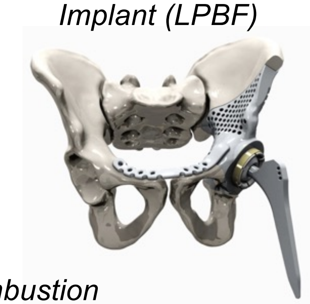
Feedstock

Printer

→ Product

Rocket engine (DED)

Implant (LPBF)



Heat exchanger (LPBF)

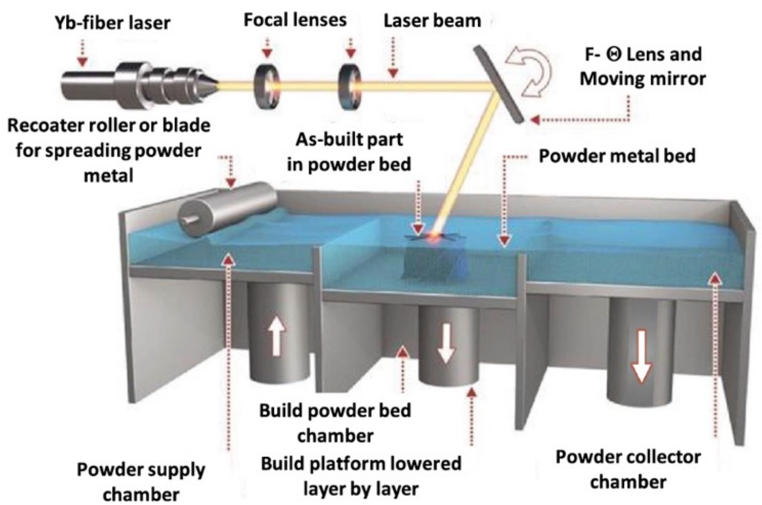
Advantages over conventional manufacturing

- Digital manufacturing nature
- Parts with complex geometries
- Highly customized components
- On site and on demand build
- Short supply chain and easy stock management
- Energy and material saving
- Multi-material build without post assembly

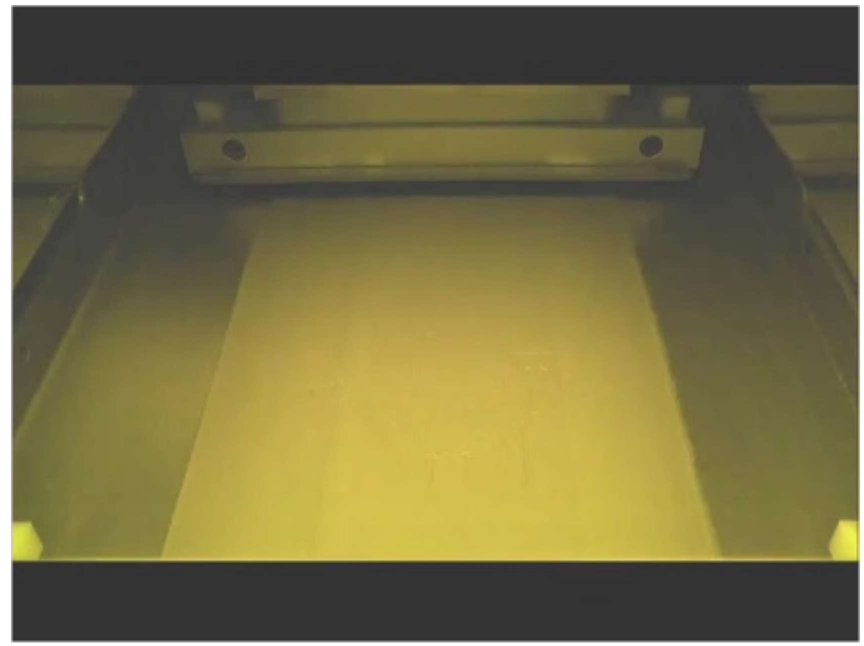
Multimaterial combustion chamber (LPBF)

Vehicle parts (BJ)

LASER POWDER BED FUSION



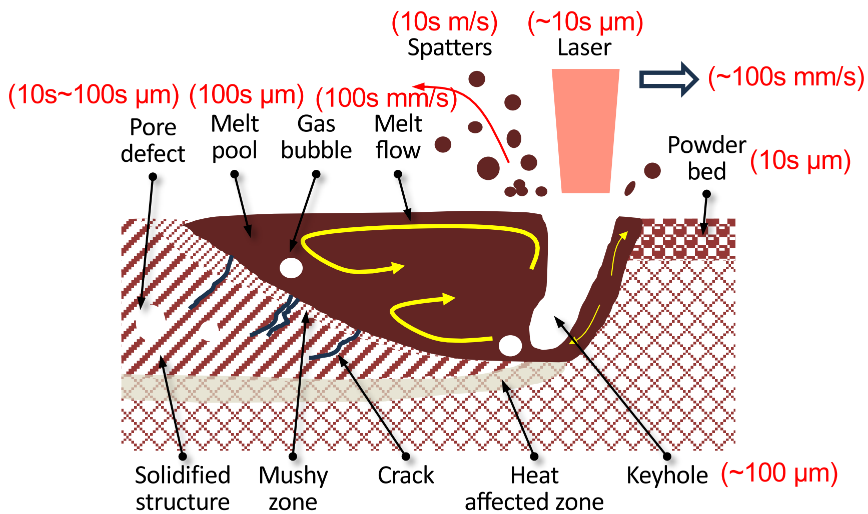
T. Özel, et al., Journal of Manufacturing Science and Engineering, 142, (2020) 011008



Advantages of laser powder bed fusion

- Complex geometries
- Fine structures

LASER POWDER BED FUSION

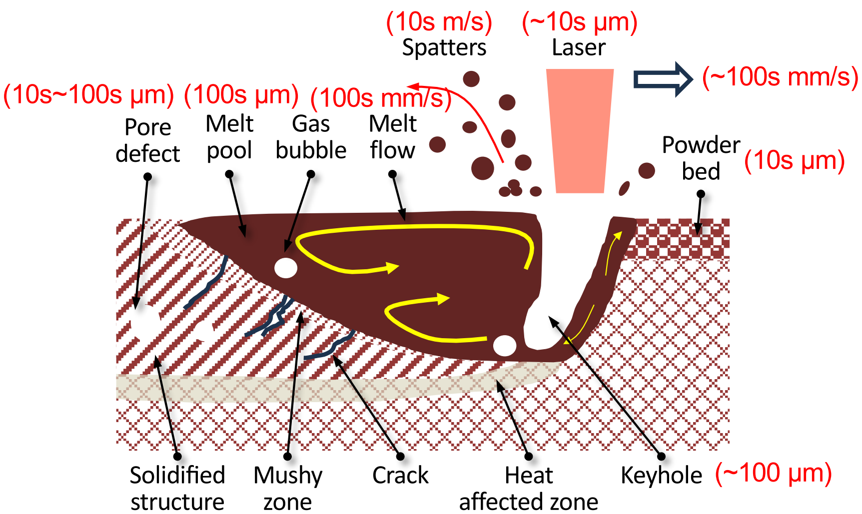


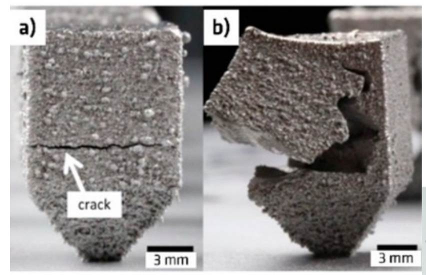
Highly dynamic phenomena

1) Dynamic laser absorption and reflection
2) Strong metal vaporization
3) Complex melt flow driven by surface tension variation and recoil pressure
4) High-velocity particle spattering driven by metal vapor
5) Powder entrainment driven by gas flow
6) Oscillation and fluctuation of keyhole
7) Rapid solidification and phase evolution

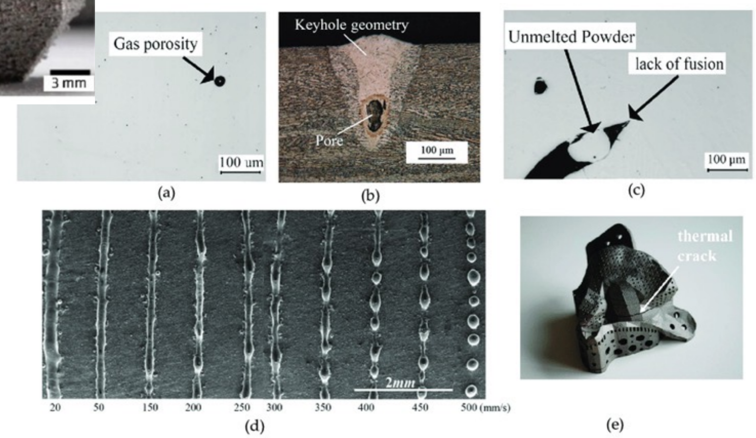
T. Sun, et al., MRS Bulletin, 45, (2020) 927
T. Sun, JOM, 72(3), (2020) 999-1008

LASER POWDER BED FUSION





Structure defects



- Dynamic multi-phase multi-force interactions
- Non-equilibrium thermal conditions
- Stochastic events

➔

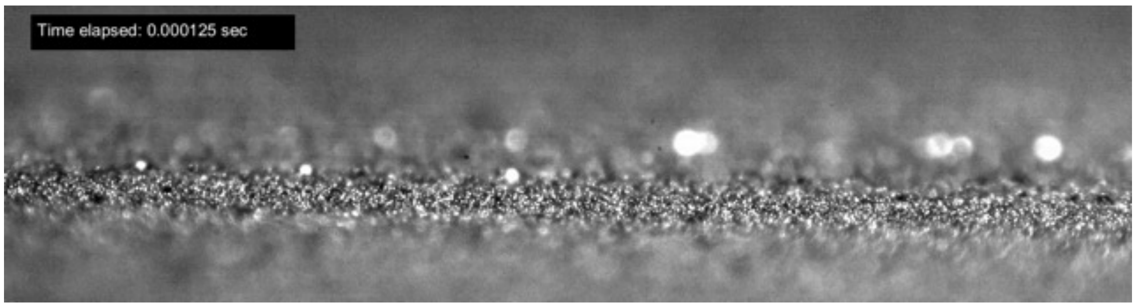
- Structure defects
- Digital twin
- Feedstock alloys
- Repeatability and reliability

➔

Qualification and certification issues

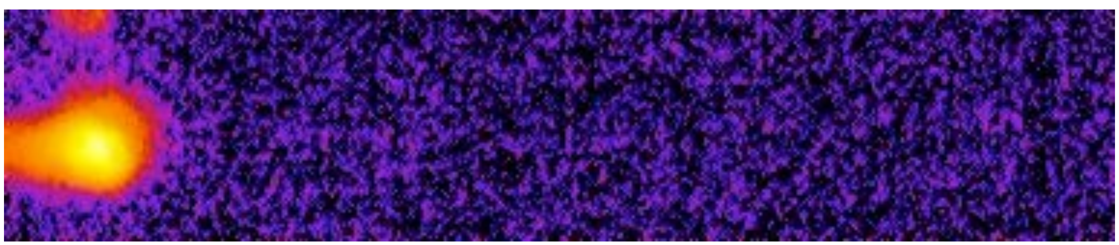
PROCESS VISUALIZATION

High-speed visible-light imaging



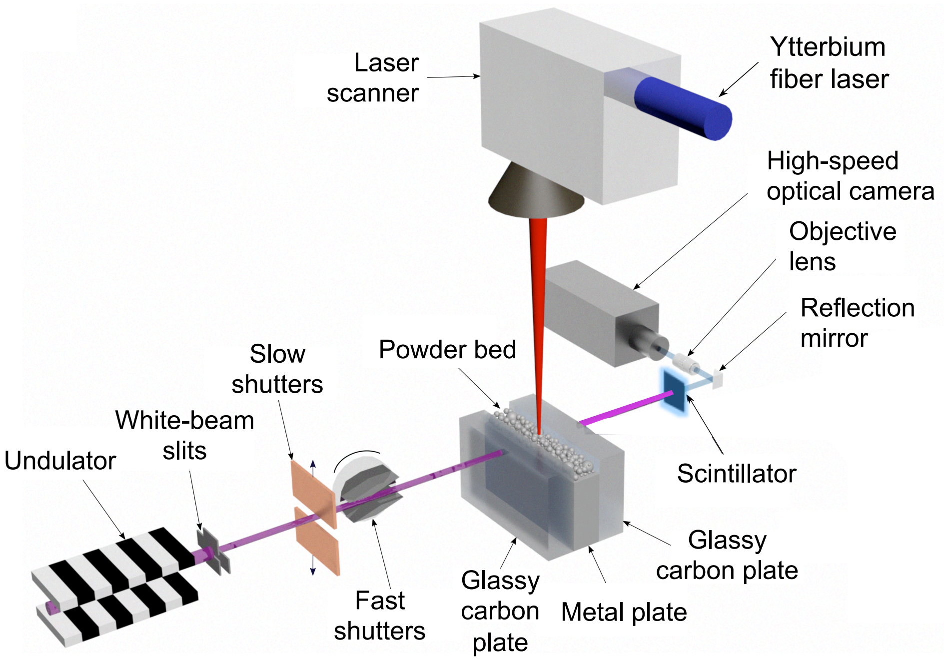
P.Bidare, et a., Acta Materialia, 142, (2018), 107-120

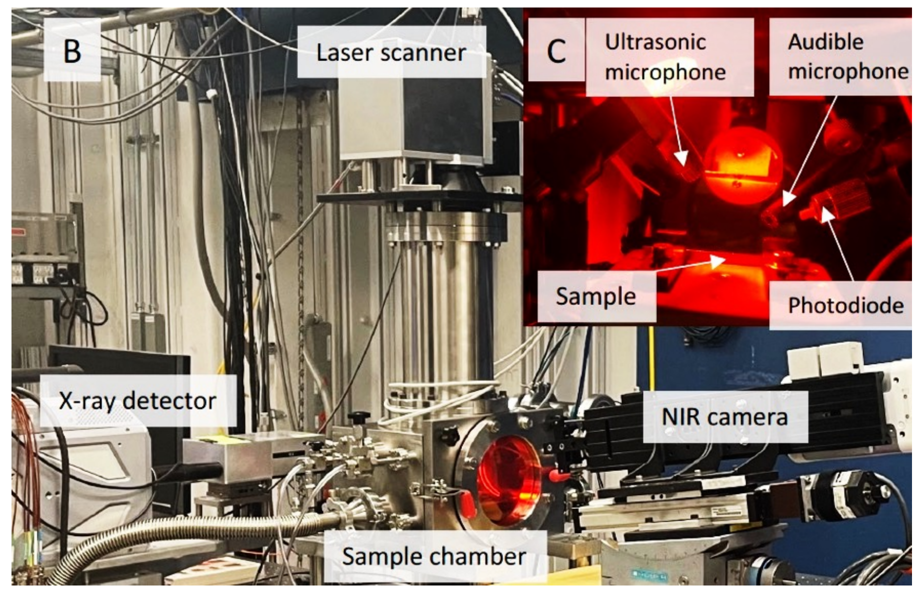
High-speed near infrared imaging



Unable to see structures below the sample surface, where most of defects are generated

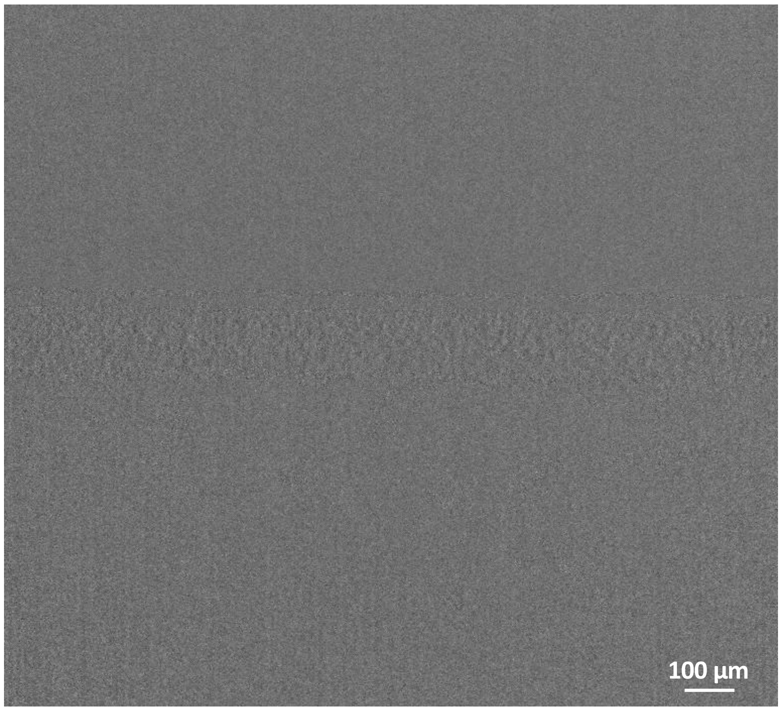
X-RAY VISION OF LASER POWDER BED FUSION





C. Zhao, et al., Scientific Reports, 7, (2017) 3602
N. Parab*, C. Zhao*, et al., Journal of Synchrotron Radiation, 25, (2018) 1467

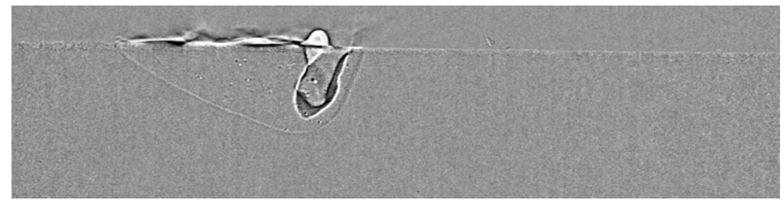
X-RAY VISION OF LASER POWDER BED FUSION



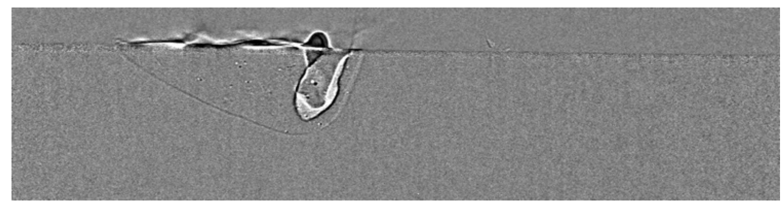
- Material: Al-10Si-Mg
- Laser power: 520 W
- Scan speed: 0.6 m/s
- Recording rate: 30,173 fps
- Exposure: 0.1 ns
- Pixel resolution: 2 µm

Simple ImageJ data processing to highlight melt pool boundary

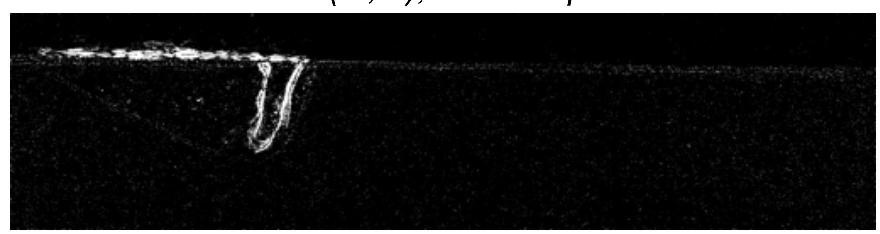
I1 = Frame 1 / Frame 2



I2 = Frame 2 / Frame 1

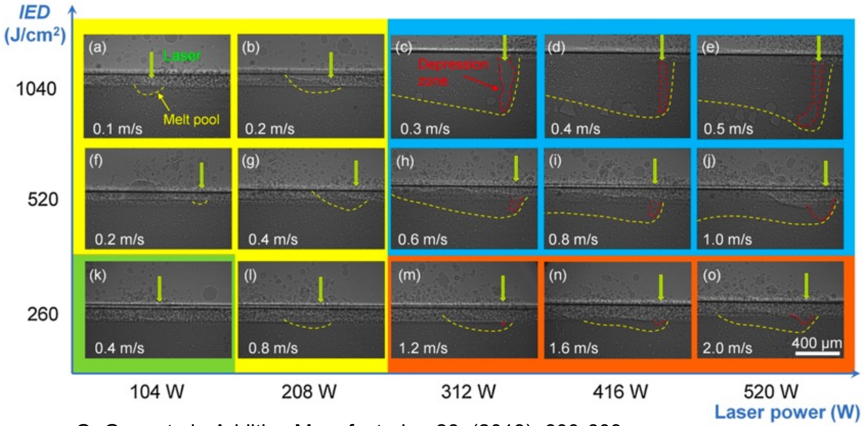


Max (I1,I2), then despeckle



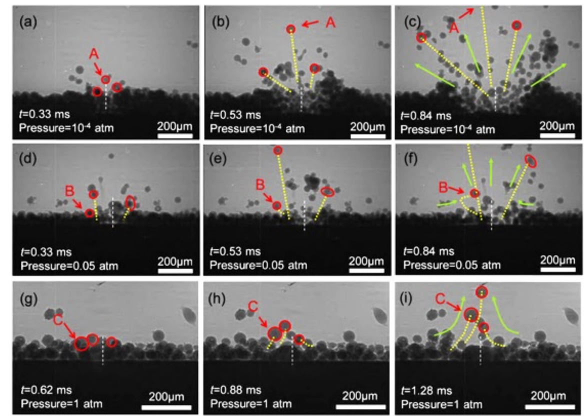
MEASURE IMPORTANT PROCESS/STRUCTURE PARAMETERS

Melt pool and keyhole morphologies



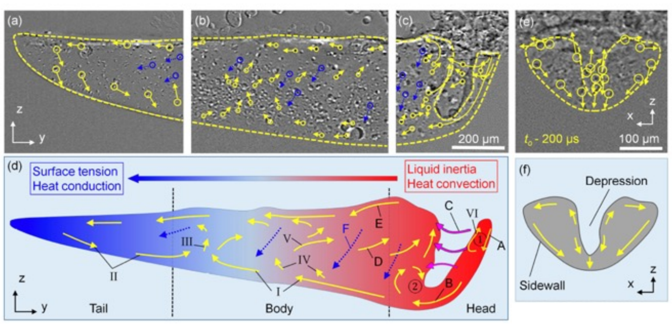
Q. Guo, et al., Additive Manufacturing 28, (2019), 600-609

Particle spattering



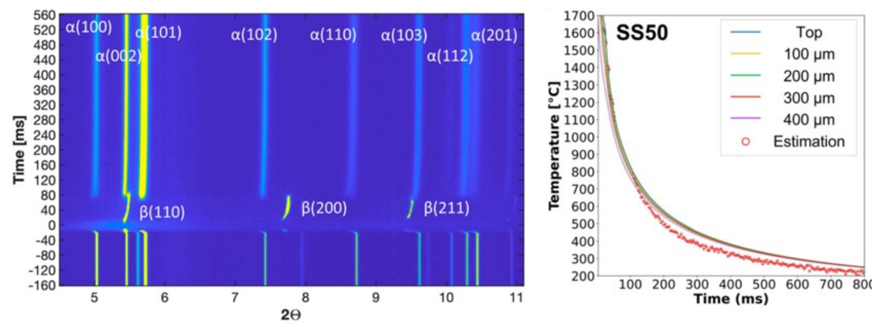
Q. Guo, C. Zhao, et al., Acta Materialia, 151, (2018) 169-180

Melt flow



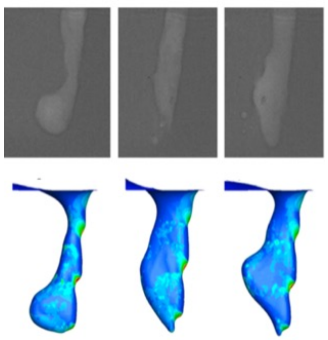
Q. Guo, et al., Additive Manufacturing, 31, (2020), 100939

Cooling rate

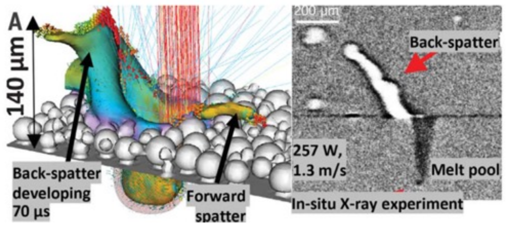


S. A. Oh, et al., Materials Research Letters, 9, (2021) 429

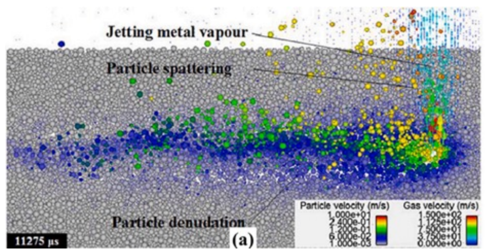
INFORM, CALIBRATE, AND VALIDATE MODELS



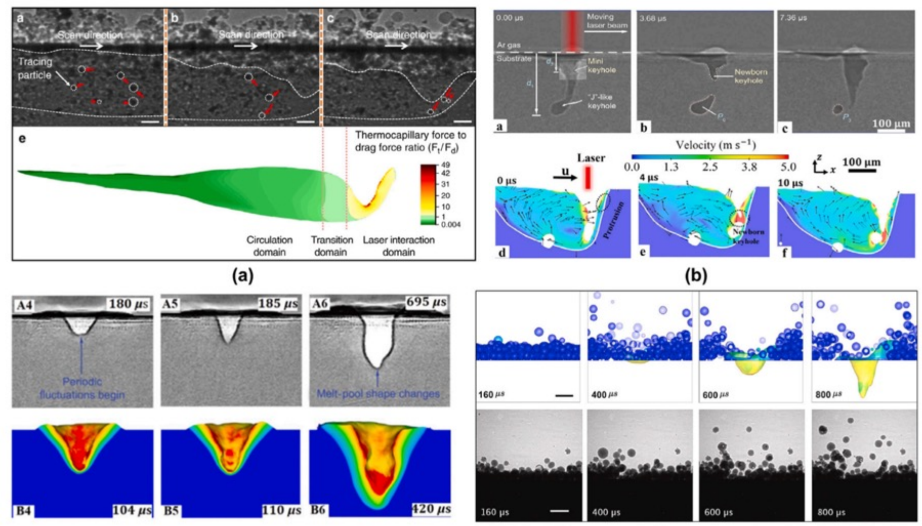
N. Kouraytem, et al., Physical Review Applied 11, (2019), 064054



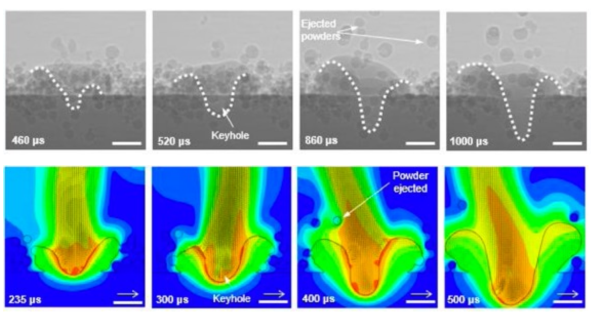
S.A. Khairallah, et al., Science, 368, (2020) 660



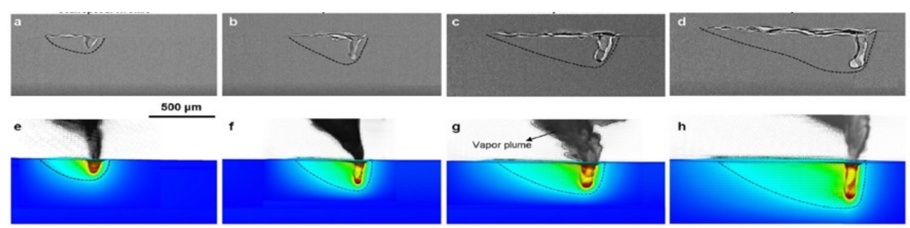
H. Chen, et al., Acta Materilia. 196, (2020) 154



L. Wang, et al., International Journal of Machine Tools and Manufacture 193 (2023) 104077



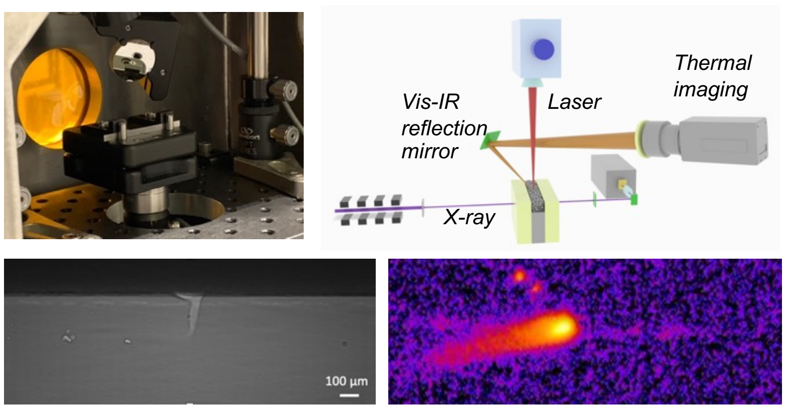
X. Li, et al., Additive Manufacturing, 35, (2020) 101362



Z. Gan, et al., Nature Communications, 12, (2021) 2379

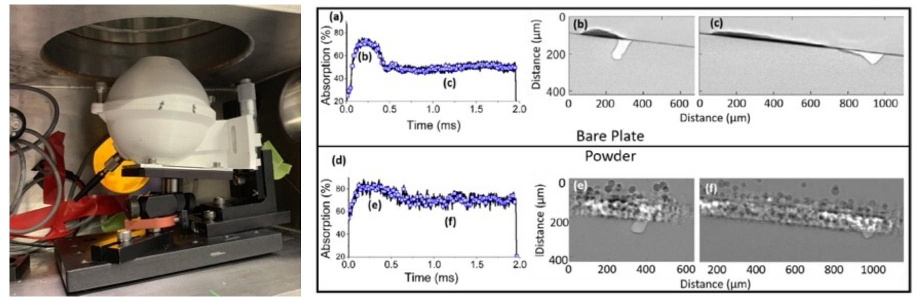
CORRELATE SENSORY SIGNALS WITH PROCESS FEATURES

Thermal imaging



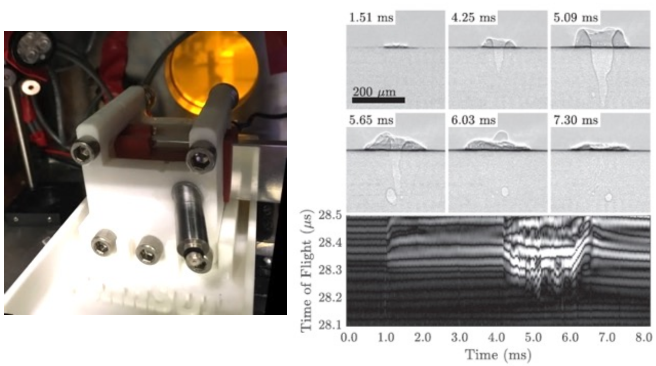
Z. Ren, et al., Science, 379, (2023) 89

Integrating sphere radiography



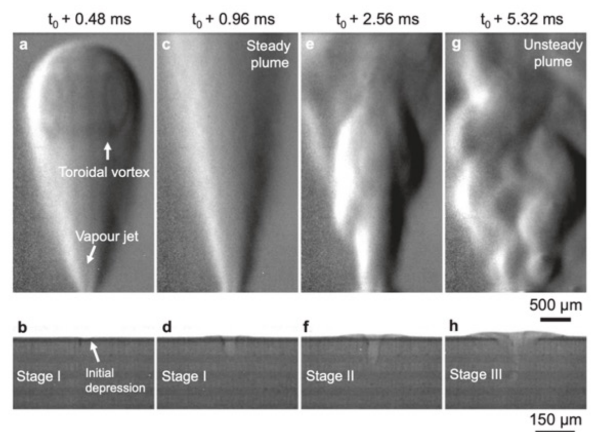
B. Simonds, et al., Applied Materials Today 23 (2021) 101049

Ultrasound



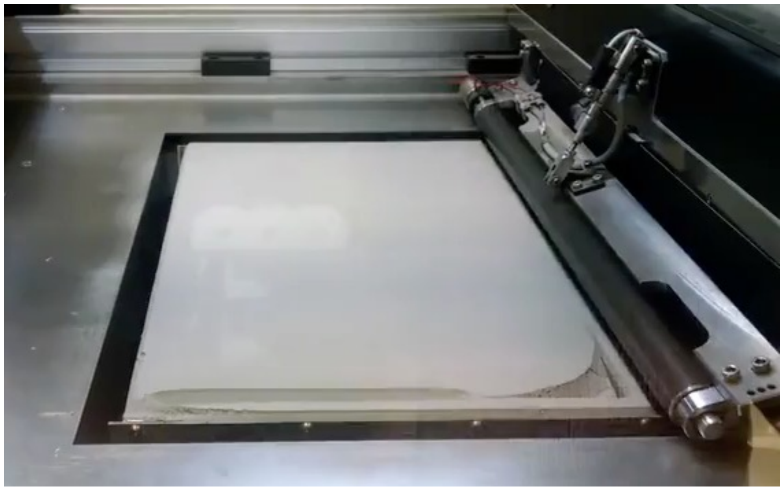
J. Gillespie, et al., Journal of Acoustic Society of America, 150, (2021) 2409

Schlieren imaging

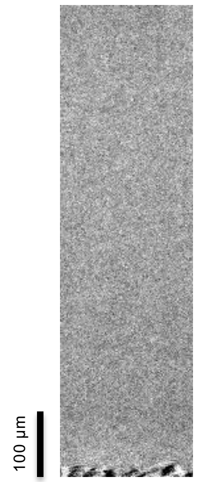


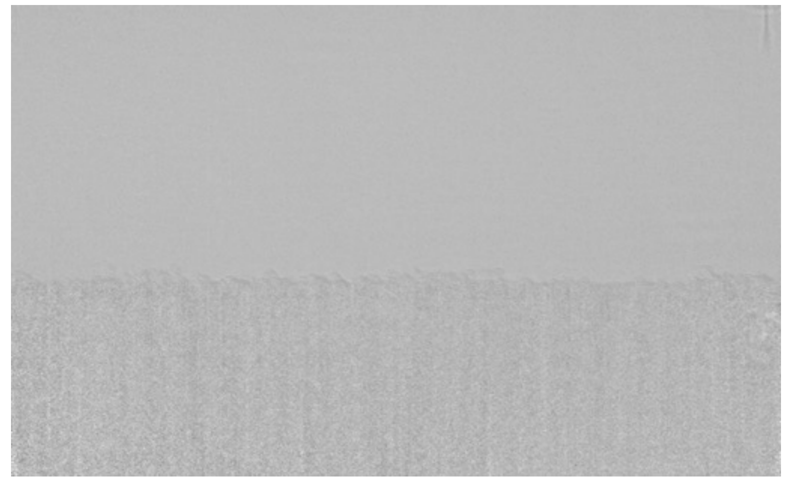
I. Bitharas, et al. Nature Communications, 13, (2022) 2959

BINDER JETTING



X-ray imaging

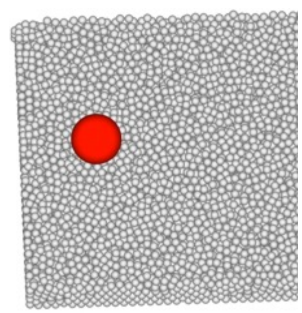


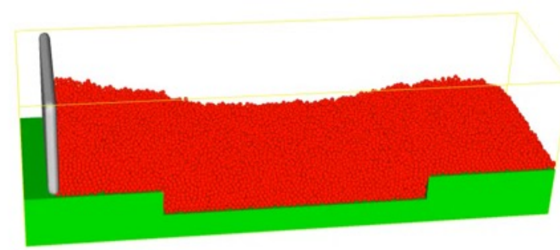


Advantages of binder jetting

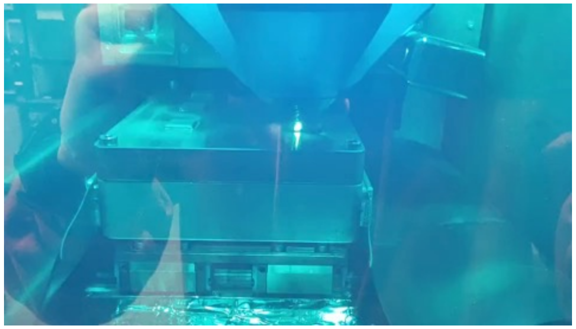
- Print → debinding → sintering
- Capable of printing all materials
- Easy to scale up for mass production
- Fine structures

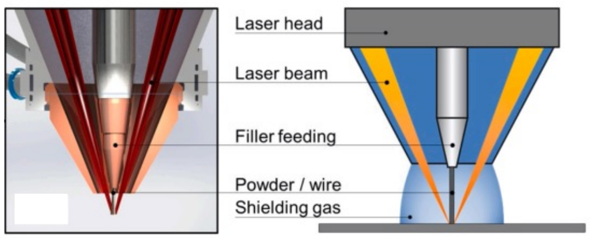
Simulations





WIRE LASER DIRECTED ENERGY DEPOSITION

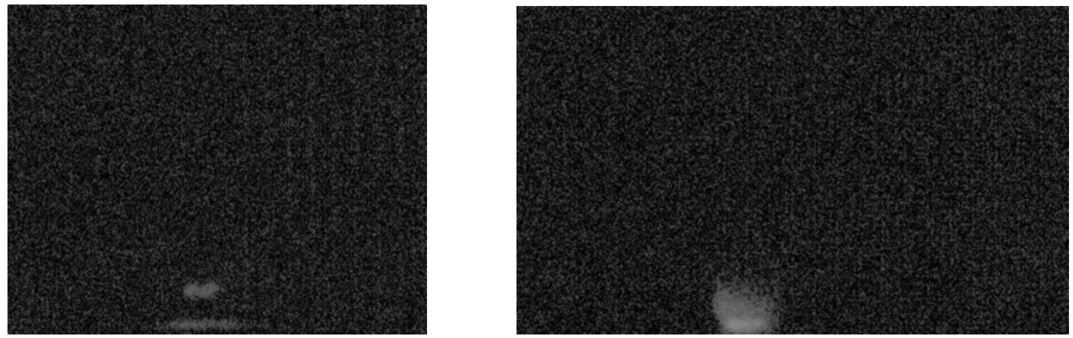




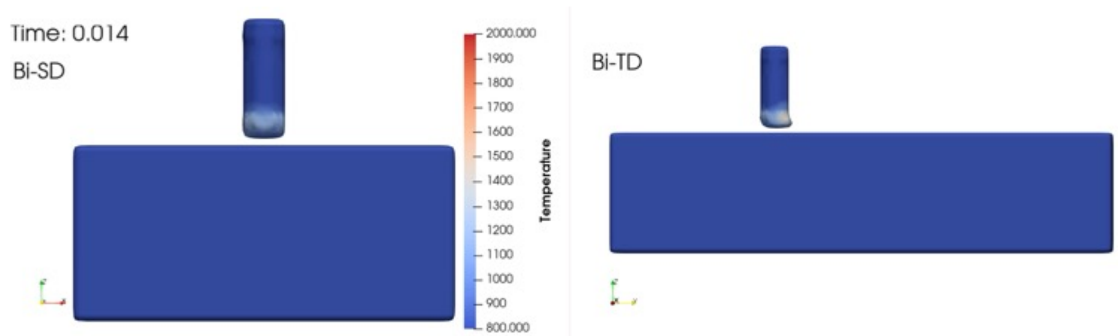
Advantages of wire-laser DED

- Large components
- Fast and efficient
- Minimum structure defects
- Minimum material waste

Optical imaging



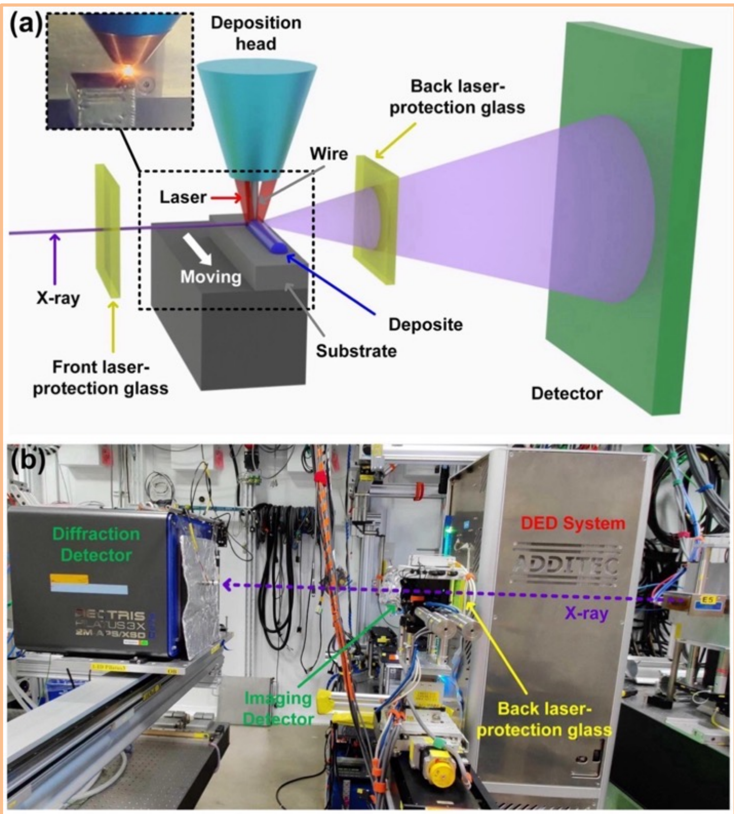
Multi-physics simulation



L. Gao, et al., International Journal of Machine Tools and Manufacture, 194, (2024) 104089
L. Gao, et al., Additive Manufacturing, 77, (2023) 103801

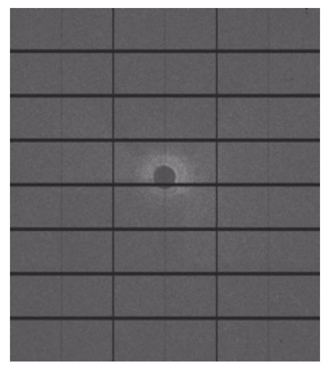
WIRE LASER DIRECTED ENERGY DEPOSITION

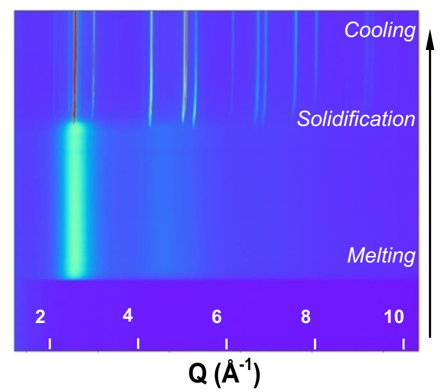
Operando synchrotron experiment



X-ray imaging

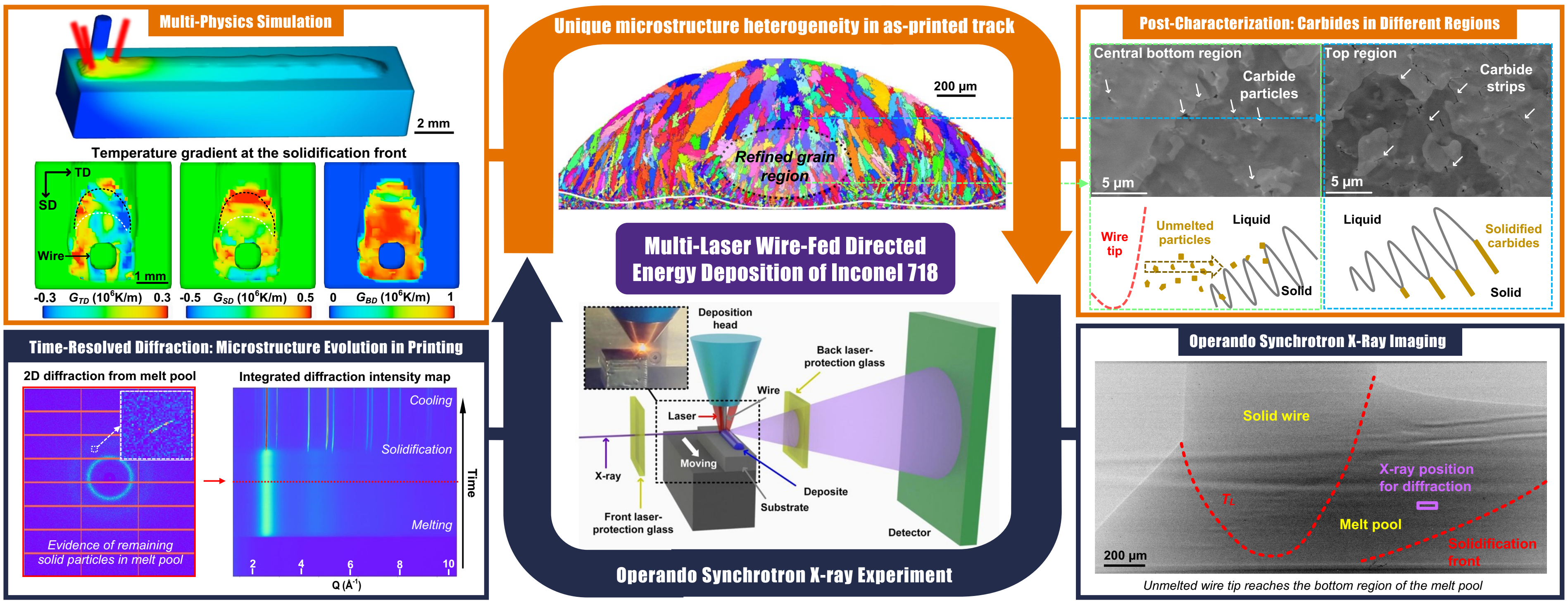
X-ray diffraction





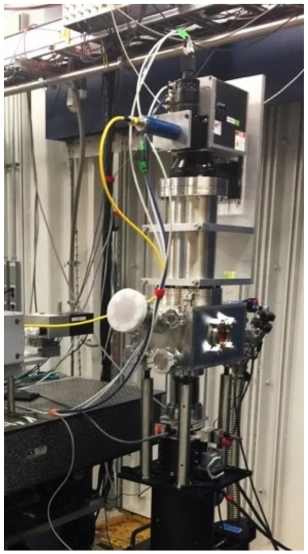
WIRE LASER DIRECTED ENERGY DEPOSITION

The operando x-ray diffraction experiment provides direct evidence that the partially melted feeding wire can reach the melt pool bottom and release solid particles near the mushy zone, which suppress the growth of large columnar grains and cause the formation of unique microstructural heterogeneity.

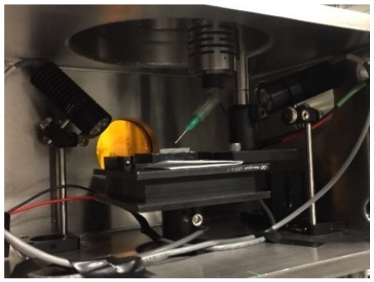


OPERANDO SYNCHROTRON EXPERIMENTS ON AM AT APS

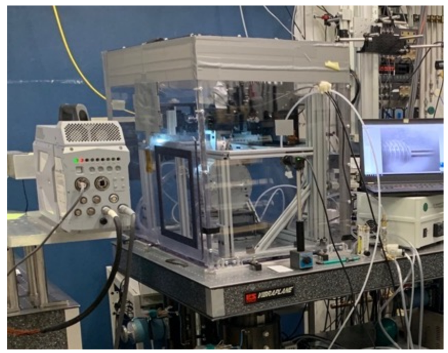
Laser powder bed fusion



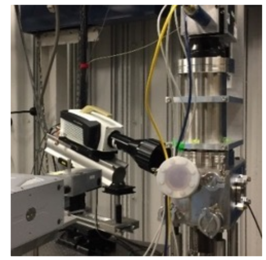
Piezo-nozzle direct energy deposition



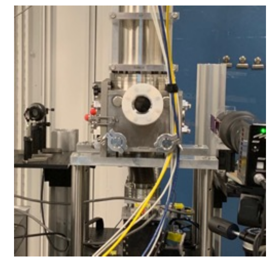
Gas-nozzle direct energy deposition



Thermal imaging

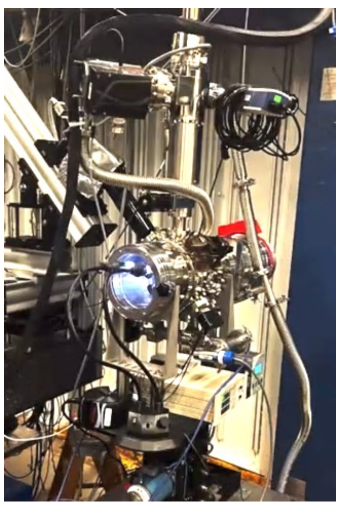


Schlieren imaging

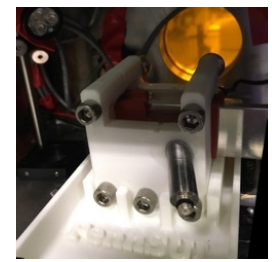


Integrated sphere

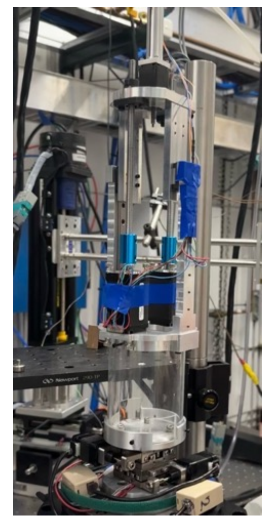
Electron beam melting



Ultrasound



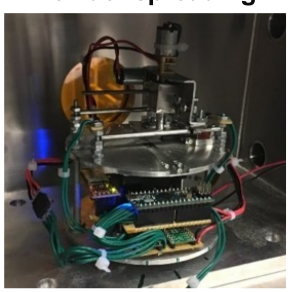
Direct ink writing



Binder jetting

Wire-laser DED

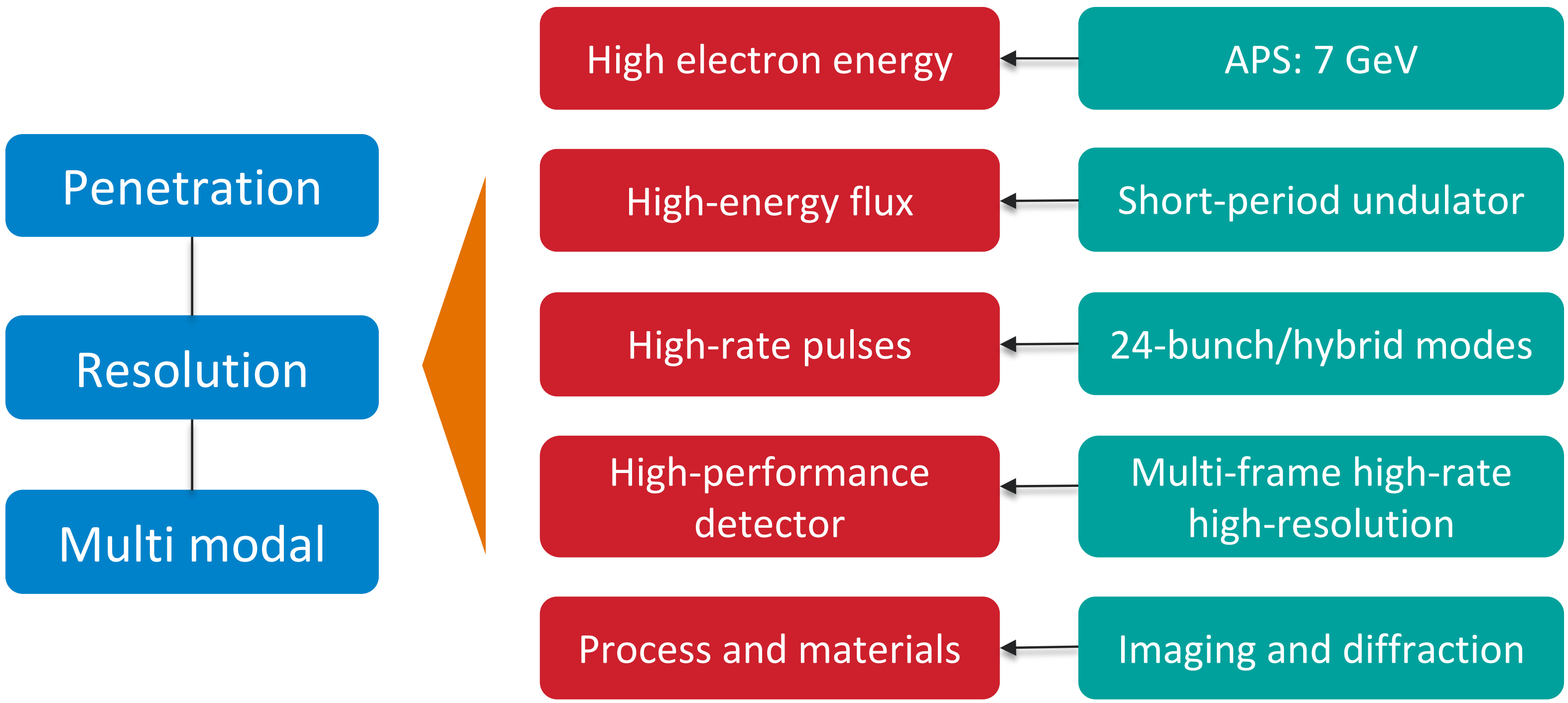
Powder spreading



Photodiode & microphone

WHY X-RAY, WHY SYNCHROTRON, WHY APS

Advanced Photon Source: 3rd-generation high-energy synchrotron facility



SYNCHROTRON VS LAB SOURCE

Advantages of synchrotron

1) **Higher brightness**
   → larger and denser samples; higher temporal resolution
2) **Smaller source and further source-to-sample distance**
   → higher spatial resolution
3) **Various pulse modes**
   → versatile time-resolved experiments
4) **Broader energy range**
   → elemental analysis; broader range of materials
5) **Advanced optics and detectors**
   → higher spatial and temporal resolutions
6) **Larger experimental hutch**
   → ample room for *in situ*/*operando* apparatus
7) **Better coherence**
   → better phase contrast; coherence-based techniques

Even better at APS-U!