

# Final Presentation

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Advanced Manufacturing  
Innovative Research Lab

**ILLINOIS TECH**

Armour College of Engineering

# Selective Laser Melting of Ti64 with HIP Post-Processing



Advanced Manufacturing  
Innovative Research Lab

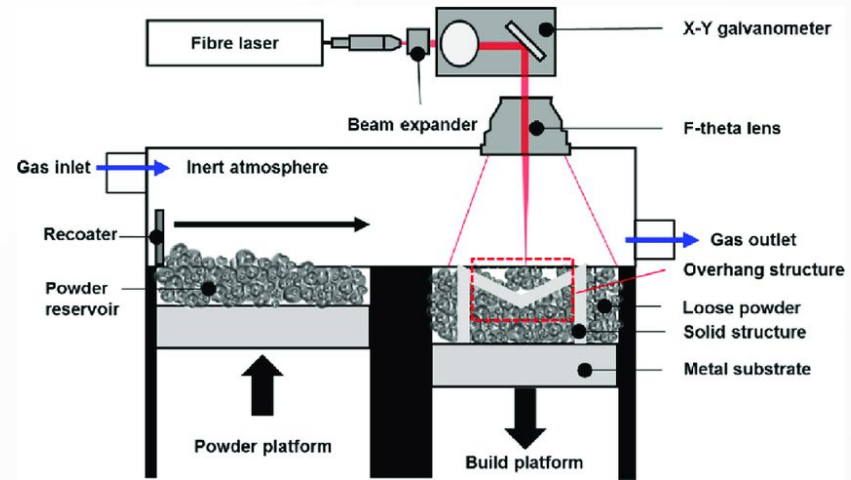
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# Brief Introduction

## What is LB-PBF (LPBF)?

- Laser beam (LB)
- Powder bed fusion (PBF)
  - an additive manufacturing process
  - thermal energy selectively fuses regions of a powder bed



## Typical LPBF machine

[https://www.researchgate.net/figure/Schematic-of-a-typical-LPBF-machine-The-build-chamber-is-purged-with-a-flowing-inert\\_fig1\\_327179759](https://www.researchgate.net/figure/Schematic-of-a-typical-LPBF-machine-The-build-chamber-is-purged-with-a-flowing-inert_fig1_327179759)

# Brief Introduction

## This work

- Ti-6Al-4V (Ti64) samples
  - $\alpha + \beta$  Ti alloy
  - Prepared using SLM printer
- Post-processing step
  - Hot isostatic pressing (HIP)
  - Close pores (apply heat and pressure at the same time)



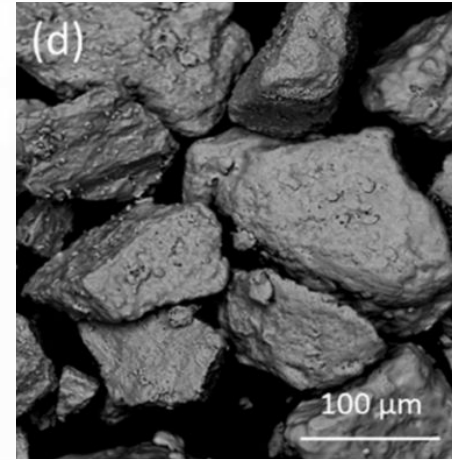
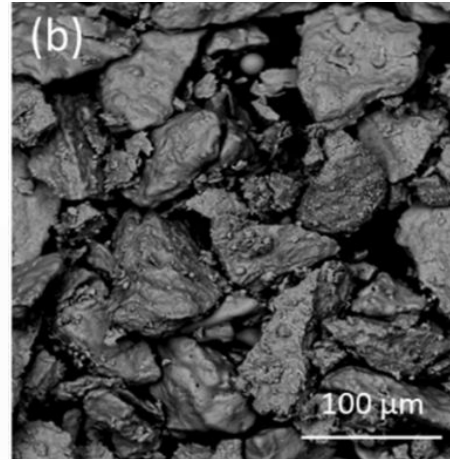
Selective laser melting printer (SLM  
Lasertec 30 DMG Mori)

<https://www.dmgmori.co.jp/en/products/machine/id=3823>

# Brief Introduction

## This work

- Hydride-dehydride (HDH) powder manufacturing process
  - Cheaper than conventional method (gas atomization)
  - Non-spherical powders
    - 50–120  $\mu\text{m}$  fine
    - 75–175  $\mu\text{m}$  coarse



(b) Fine powder, (d) Coarse powder

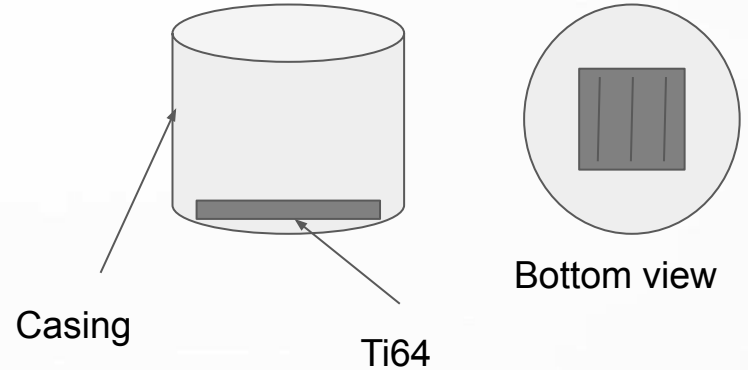
# Sample Preparation - Grinding & Polishing

## Goal

- Remove surface topography to have flat surface for characterization (e.g., optical microscopy)

## Method (Grinding)

- Water constantly flowing onto sandpaper
  - Rotating wheel or stationary
- Rotate sample 90° with each paper to remove lines
  - Grits: 240, 400, 600, 800, 1200



Ti64 sample in polymer mold (after grinding with 240 grit)

# Sample Preparation - Grinding & Polishing

## Method (Polishing)

- Optical microscopy
  - Polishing cloth on rotating wheel
    - 3  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  slurry (~10 min)
    - 1  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  slurry (~7 min)
  - Vibrating polisher
    - 0.05  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  slurry (~30 min)



Polished sample,  
surface is mirror-like

# Sample Preparation - Grinding & Polishing

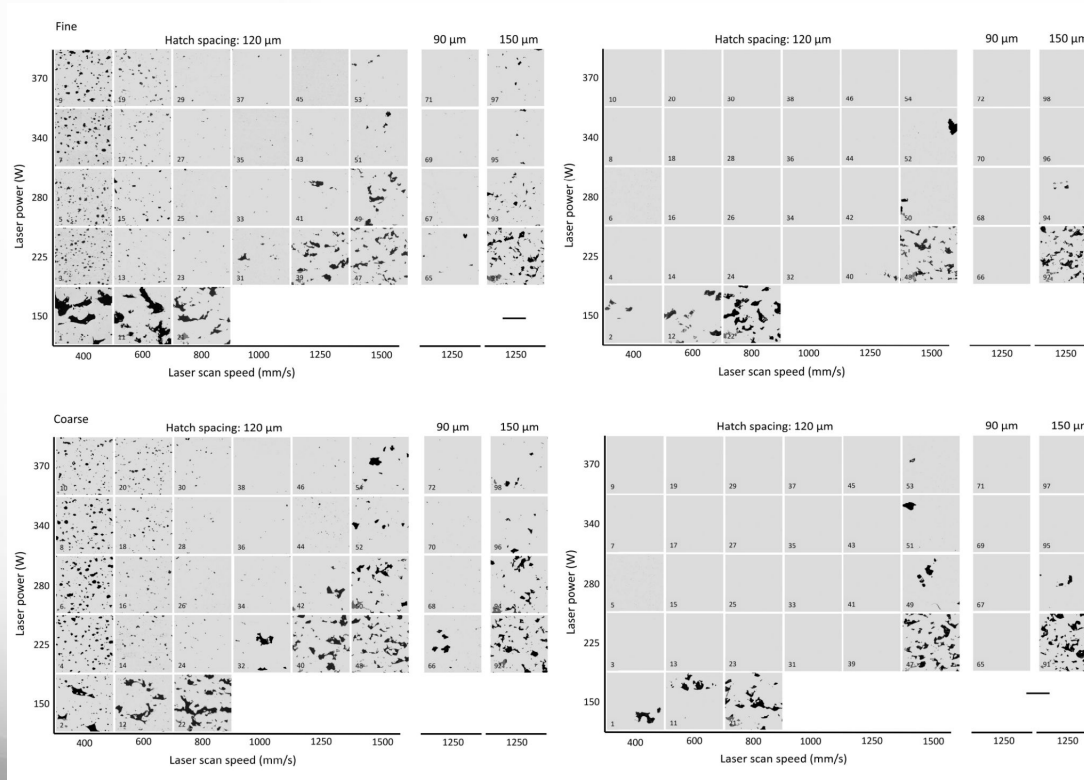
## Method (Polishing)

- EBSD
  - Add P4000 grinding paper
  - Polishing cloth on rotating wheel
    - 1  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  slurry (~7 min)
  - Vibrating polisher
    - 0.05  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  slurry (1 h 15 min)
  - Colloidal silica polisher
    - 0.04  $\mu\text{m}$  colloidal  $\text{SiO}_2$  (1 h 39 min)



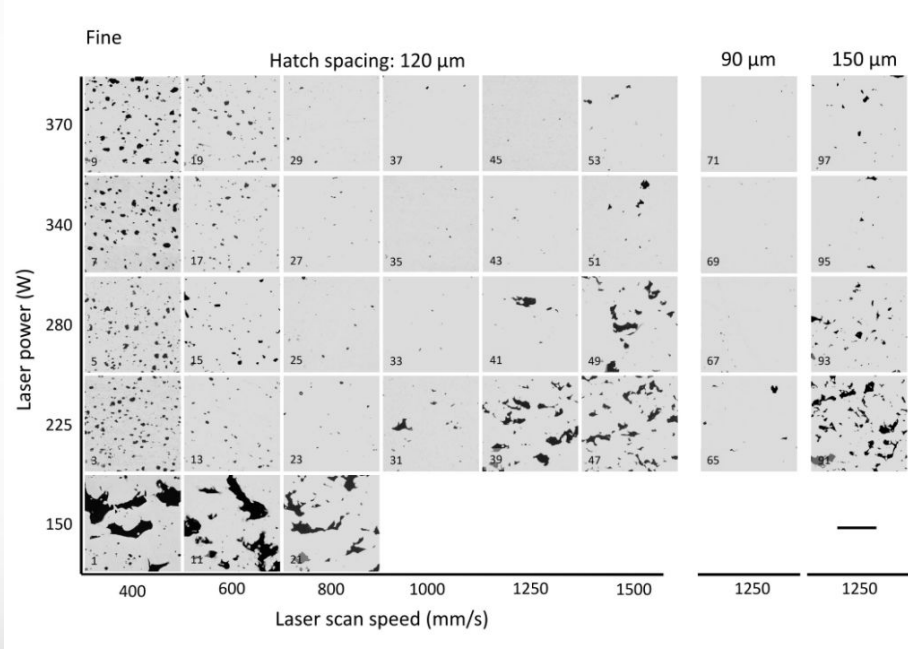
Machine for polishing with colloidal silica

# Ti64 Processing Map (Before and After HIP)

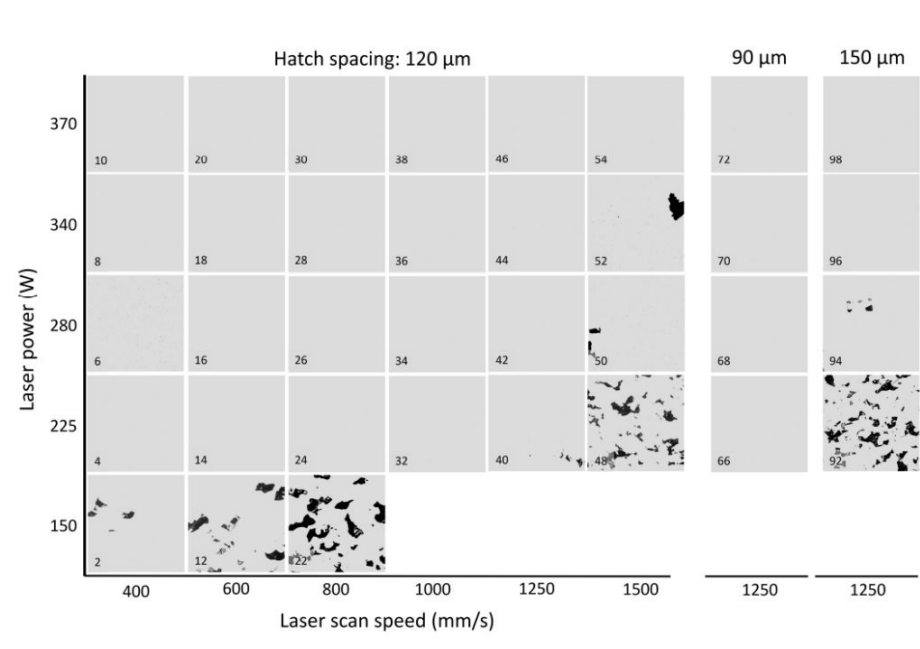


Processing parameter map. The left side is before HIP treatment, and the right side is after.

# Ti64 Processing Map



Fine powder samples before HIP



Fine powder samples after HIP. Smaller and less frequent pores

# Hardness Test (Theory)

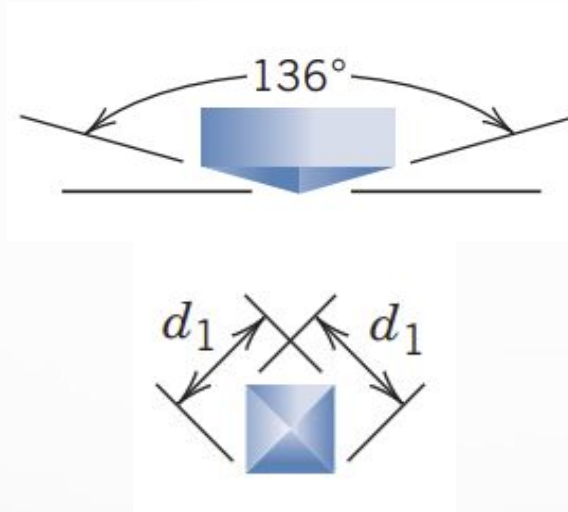


Illustration of Vickers hardness indent

$$HV = 1.854P/d_1^2$$

Vickers Hardness Equation

- P: applied load (kg)
- $d_1$ : diagonal (mm)

# Hardness Test

## Goal

- Measure hardness (resistance to permanent deformation) of Ti64 samples
  - Fine powder, 120  $\mu\text{m}$  hatch spacing

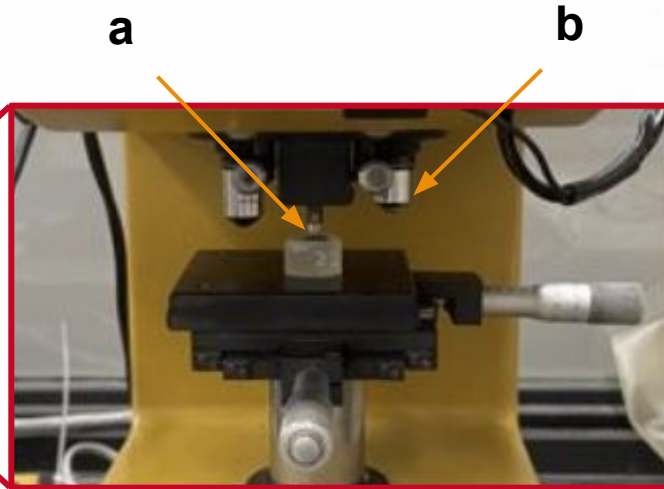
## Method

- Indent sample 12 times
  - 3 rows of 4
  - Avoid pores, other indents, and epoxy
- Measure diagonals using optical microscope
  - Not necessarily equal
- Vickers Hardness test
  - Can be converted into Pa

# Hardness Indentation



Digital Micro Hardness Tester



(a) Diamond tip indenter, (b) Lens

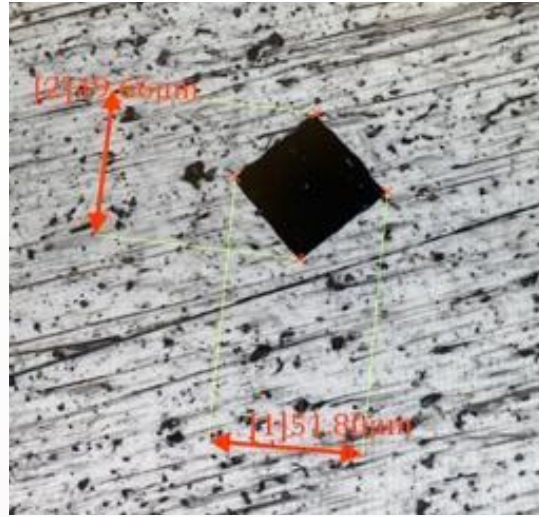
## Test Parameters

- 15 s load time
- 500 g load

# Hardness Measurements



Optical microscope



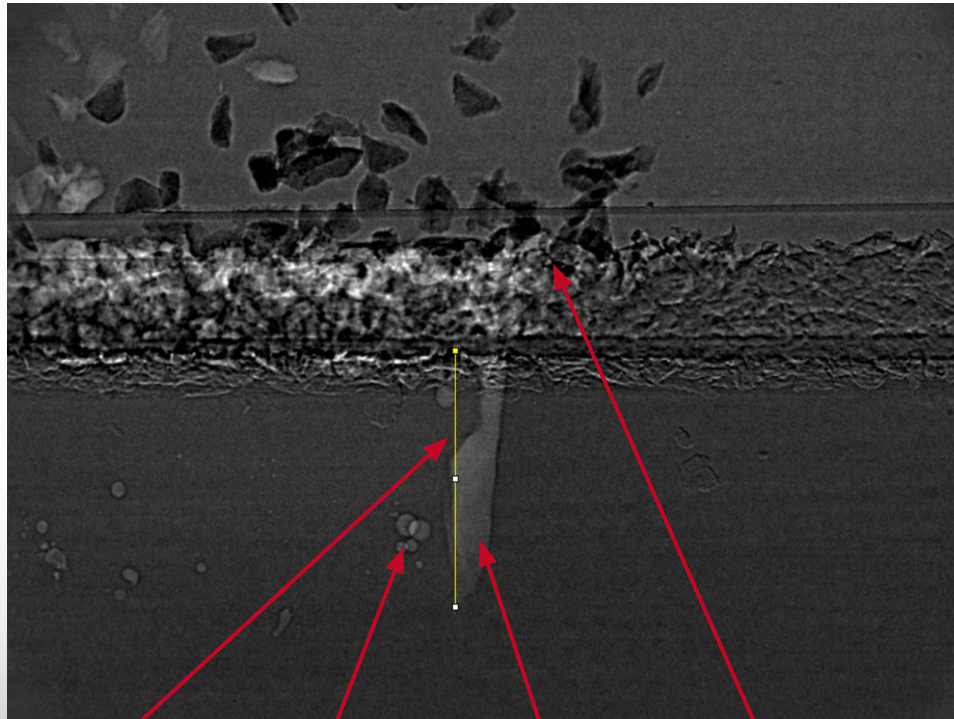
Measure diagonals  
(computer accounts for  
magnification)

**Total Average** (across all  
samples)

358.83 HV<sub>0.5</sub>

- Similar value to LPBF Ti64 w/o HIP
- Reference for Vickers:
  - WC ~2500
  - Epoxy ~45
  - LPBF 316L SS ~240

# Dynamic X-ray Radiography Measurements

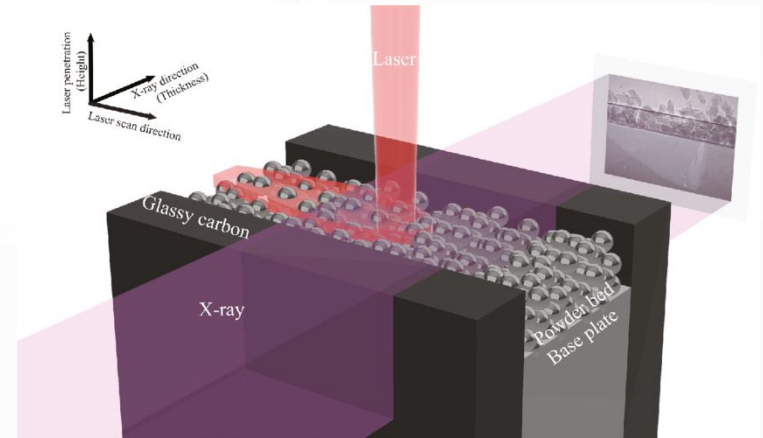


1

2

3

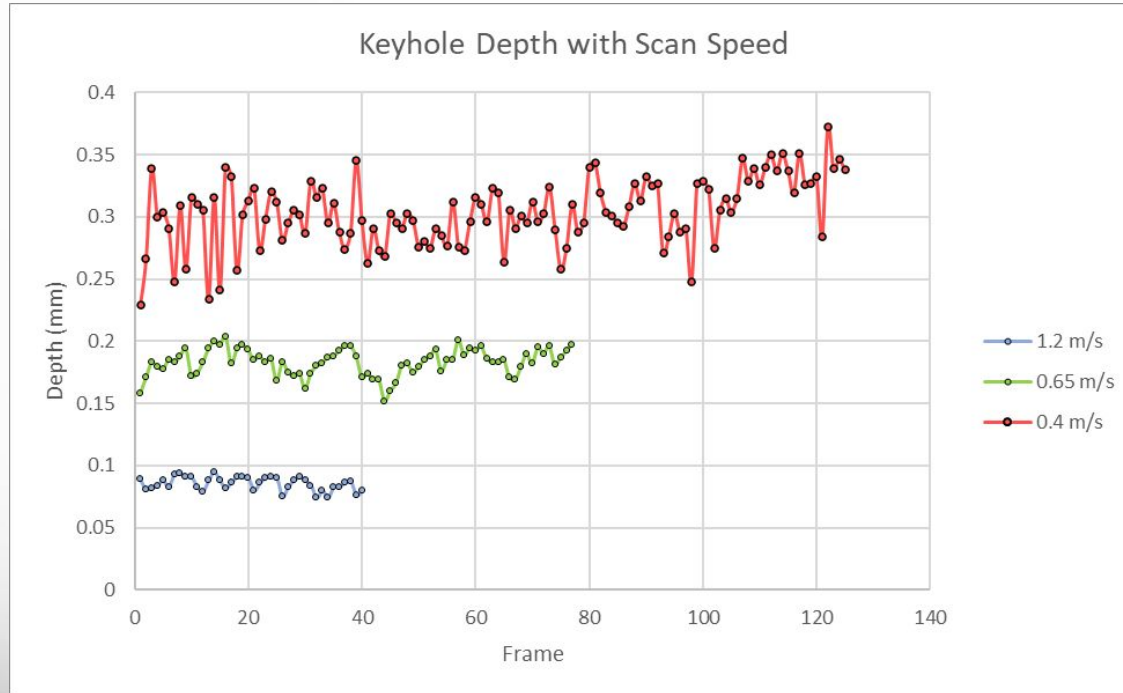
4



Schematic of setup at APS

- 1) Measuring tool
- 2) Keyhole pores
- 3) Keyhole
- 4) Powder layer and spattering

# Dynamic X-ray Radiography Results



Speed (m/s)	Avg Depth (mm)	Stdev (mm)
0.4	0.3037	0.02677
0.65	0.1831	0.01060
1.2	0.0855	0.00545

# Accomplishments

## Measured

- Surface roughness
- Hardness
- Fatigue life
- Keyhole porosity depth from dynamic x-ray radiography images

## Collected

- Optical microscopy images
  - Polished surface
  - Surface roughness “heat” map
- Porosity data from CT scan

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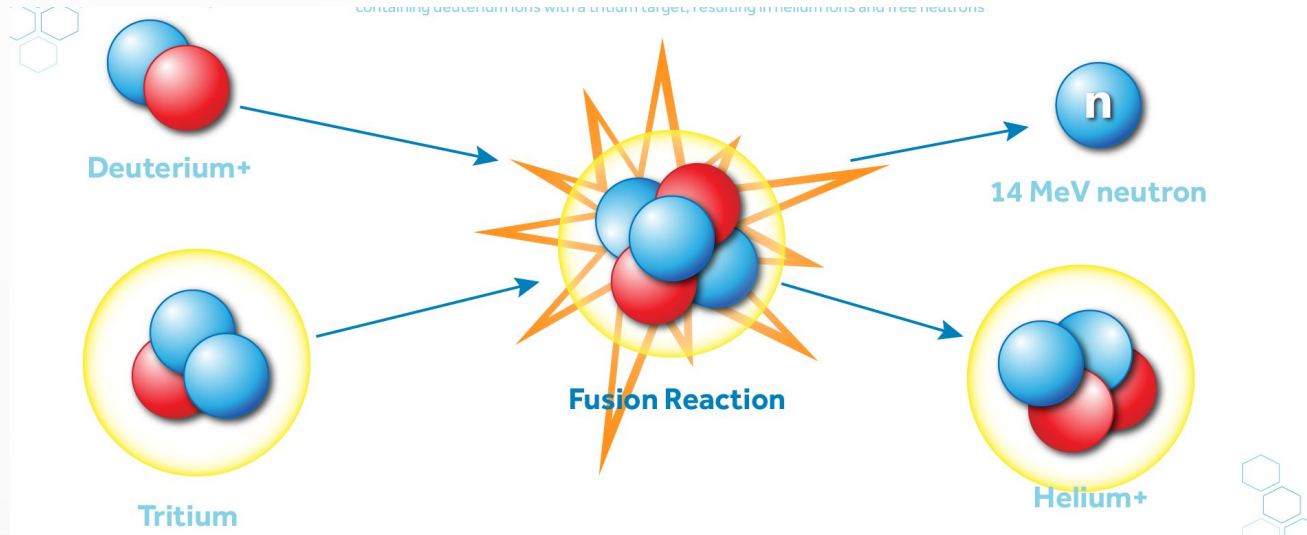
# Combined Neutron and X-ray Imaging Literature Review and Potential Commercial Application



# Brief History

- 1932** James Chadwick discovered the neutron
- 1935-44** First reported use of neutron activation analysis (NAA)
- Neutrons hit object, radioactive isotopes form in object, gamma rays emitted are measured using film (now digital)
- 1943** First usable nuclear reactor (at Oak Ridge) was built
- 1950s** Availability of small accelerator-based neutron generators (increased use of neutrons in laboratories)
- Late 1950s** Geologists and petroleum engineers used portable neutron generators (for fast NAA (FNAA))
- 70s - Present** Facilities built and new techniques developed

# Neutron Generation



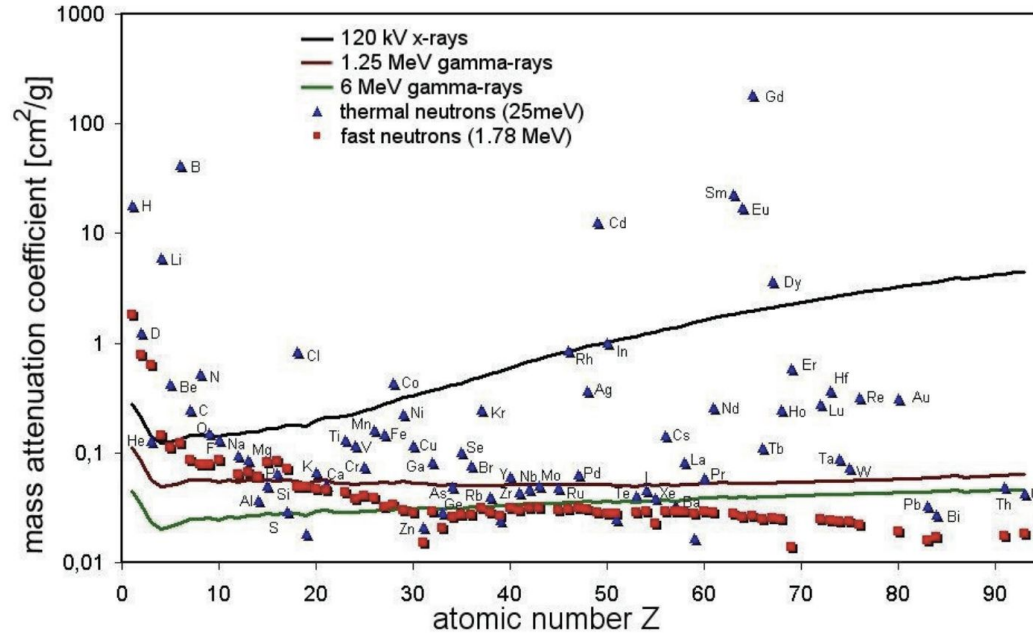
D = Deuterium  
T = Tritium

Imaging (Most systems)	
Cold	Thermal
< 25 meV	25 meV

- DT fusion
  - Deuterium ion beam strikes target loaded with tritium
  - Produces 14.1 MeV neutron
- Also DD fusion
  - Produces 2.45 MeV neutron

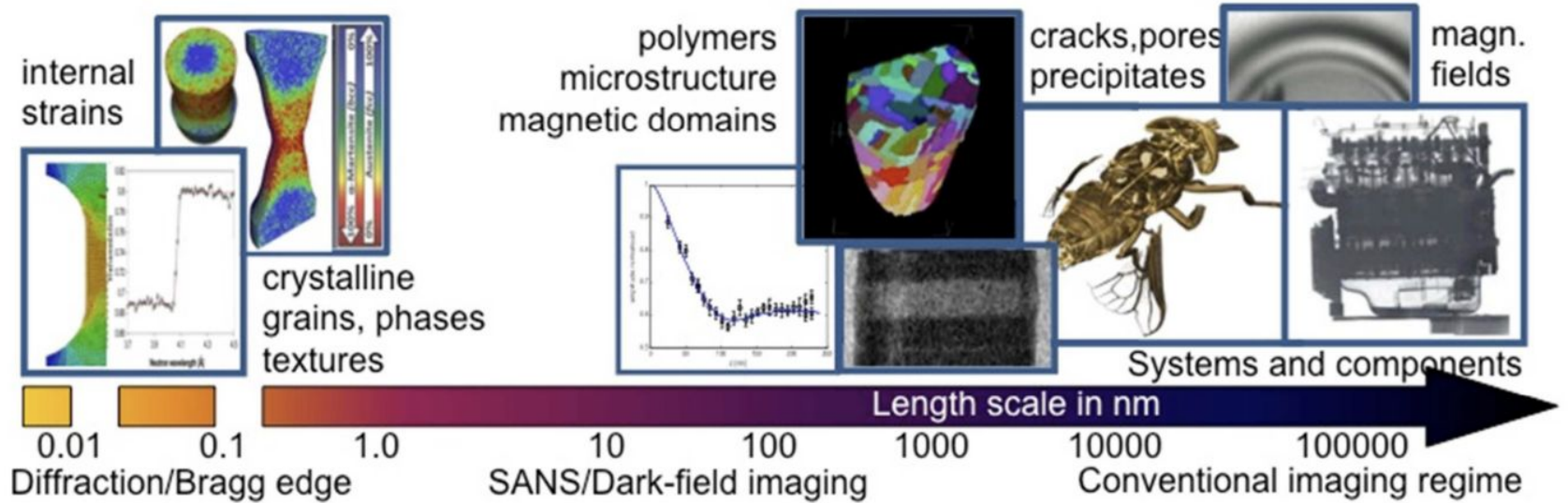
<https://www.shinefusion.com/neutron-generators/>

# Attenuation vs. Z



- X-ray attenuation increases with Z
- Thermal neutrons show no simple relationship, while fast neutrons decrease with Z
- Note that cold neutrons are  $< 25$  meV

# Modern Neutron Imaging Capabilities



Map of neutron imaging capabilities, from material data to images of large components

# Neutron and X-ray Imaging (Lab)

- Simultaneous neutron and x-ray imaging
  - ICON at PSI, Switzerland
  - NeXT at ILL Grenoble, France
  - NeXT at NIST, USA
  - ODIN at ESS, Sweden
  - VENUS at ORNL, USA
- Neutron and x-ray imaging, not simultaneous
  - NEUTRA at PSI, Switzerland
    - Plans to upgrade XTRA option at NEUTRA to enable simultaneous bi-modal neutron and x-ray imaging

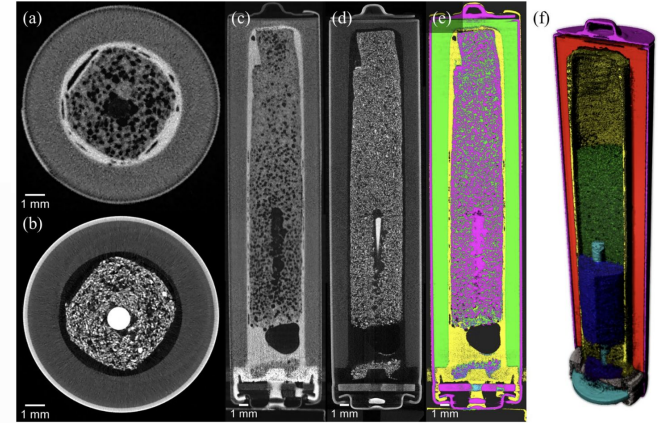
# Combined Neutron and X-ray Imaging

## Published

- Imaging composite materials
  - Volume segmentation of batteries and cement
  - Soil, electronic components, corroded nails, artifacts (e.g., sword), etc.

## Not Published (as far as I know, possibly by 2023)

- Additive manufacturing
  - Some papers using neutron tomography



Neutrons pick up polymer separator, while x-rays show current collector

# Simultaneous Neutron/X-ray Scan

## N source/detector

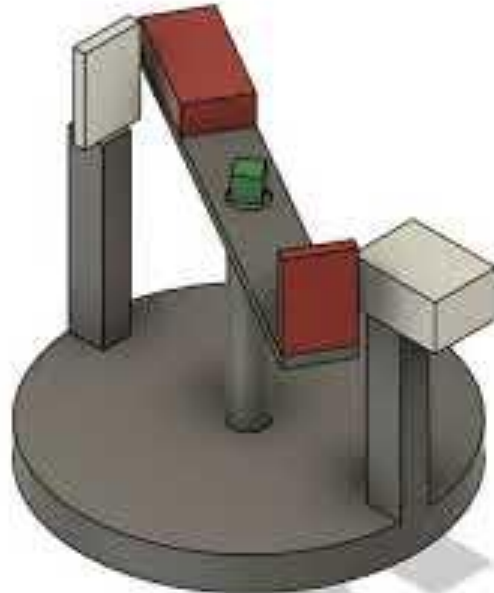
- rotate  $60^\circ$
- wait  $90^\circ$
- rotate  $60^\circ$

## Sample

- rotate  $360^\circ$

## X-ray source/detector

- stationary



# Future Work

- Continue researching sources
- Figure out what needs to be done commercially, and why?
- Answer: Do we have  $n$  sources for tomography? If not, for imaging? If not,  $n$  source/detector move with the sample.
- Figure out energy/exposure required for neutron images
  - For  $4 \times 10^7$  n/s output, image took 4 hours (first radiography, 1935)
  - 1946: Neutron radiograph took a few minutes
  - What are the times nowadays?

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