

AM Defect Detection Using X-ray Phase Contrast Imaging

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*Exceptional
service
in the
national
interest*

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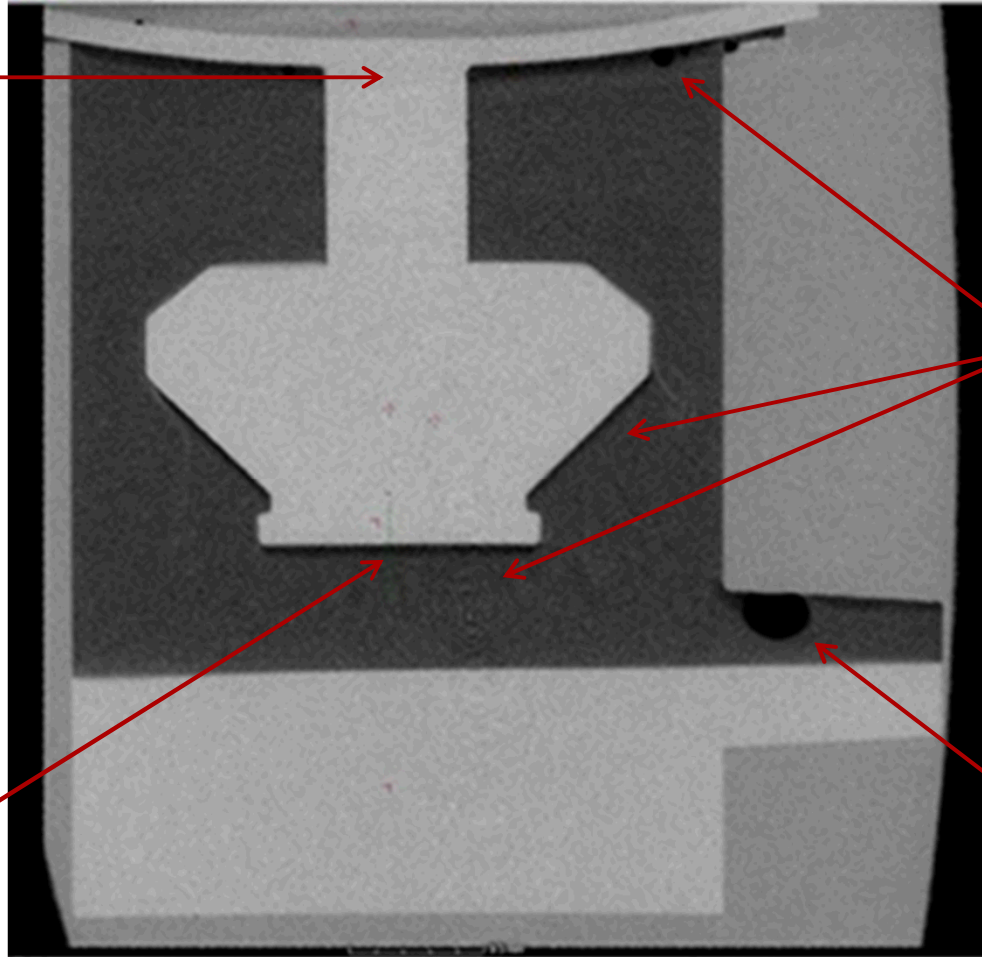
- Defect detection and validation & verification in AM
- Introduction to X-ray phase contrast imaging
- Application to defect detection for AM
- Status at Sandia-NM

DEFECT DETECTION AND VALIDATION & VERIFICATION IN AM

- Inspection is critical to assuring functionality and security
 - “Slice and dice” inspection
 - Randomly selected samples
 - Labor intensive
 - Limited imaging depth
 - Complicated sample preparation
 - Limited in materials that can be inspected
- X-ray phase contrast imaging (XPCI)
 - Non-destructive
 - Three orders of magnitude greater sensitivity to phase over absorption imaging
 - Complementary imaging modalities (absorption, phase, dark-field)
 - Visualization of internal structure
 - Compare part topography to CAD design
 - Look for cracks, voids
 - Identify unfused powder

AM Part Imaged at SNL Using X-ray CT

Suspending
beam bows
from shrinkage



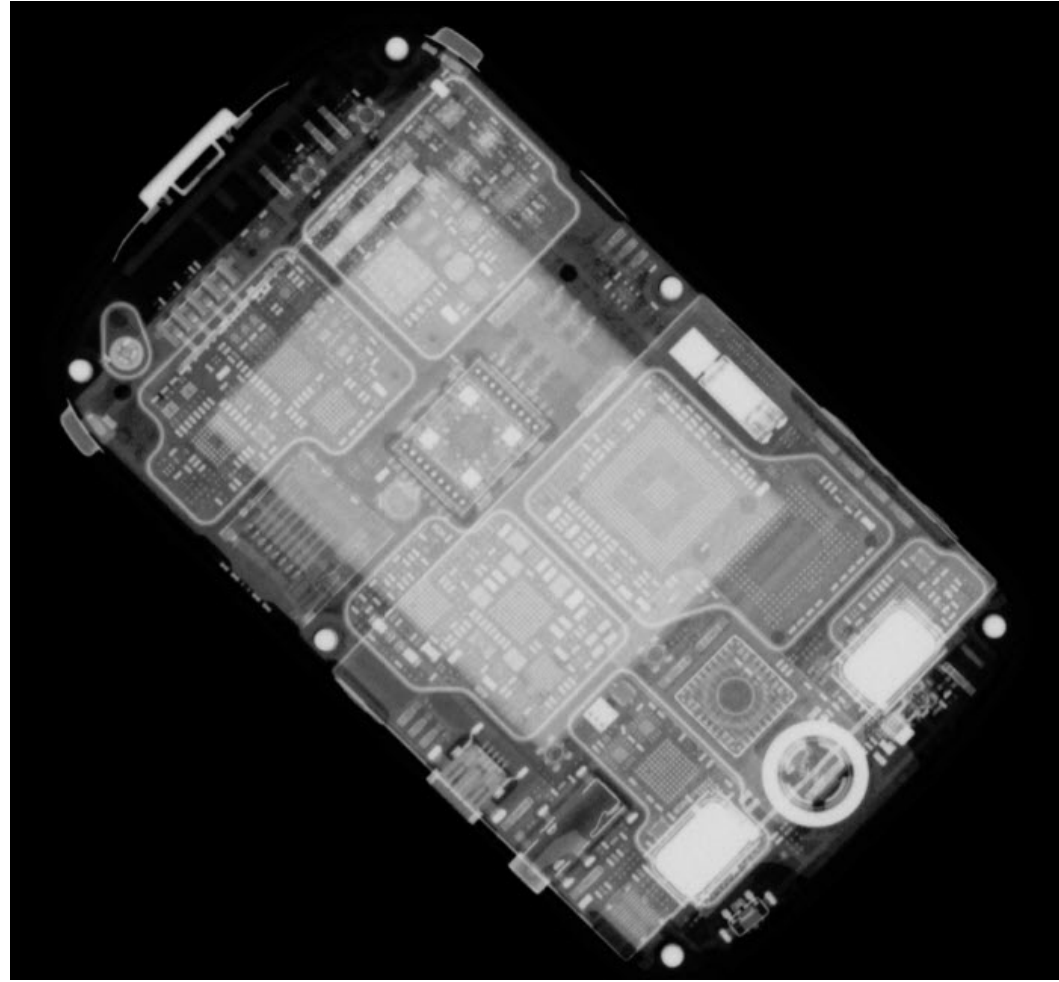
Lower density regions

Void from
delamination of
epoxy from shape

Large bubble
causes void

INTRODUCTION TO X-RAY PHASE CONTRAST IMAGING

X-ray Phase Contrast Imaging





Absorption

Contrast between dense and less-dense (low-Z) regions.
No material detail.



Phase Contrast

Fine structure within the low-Z regions.



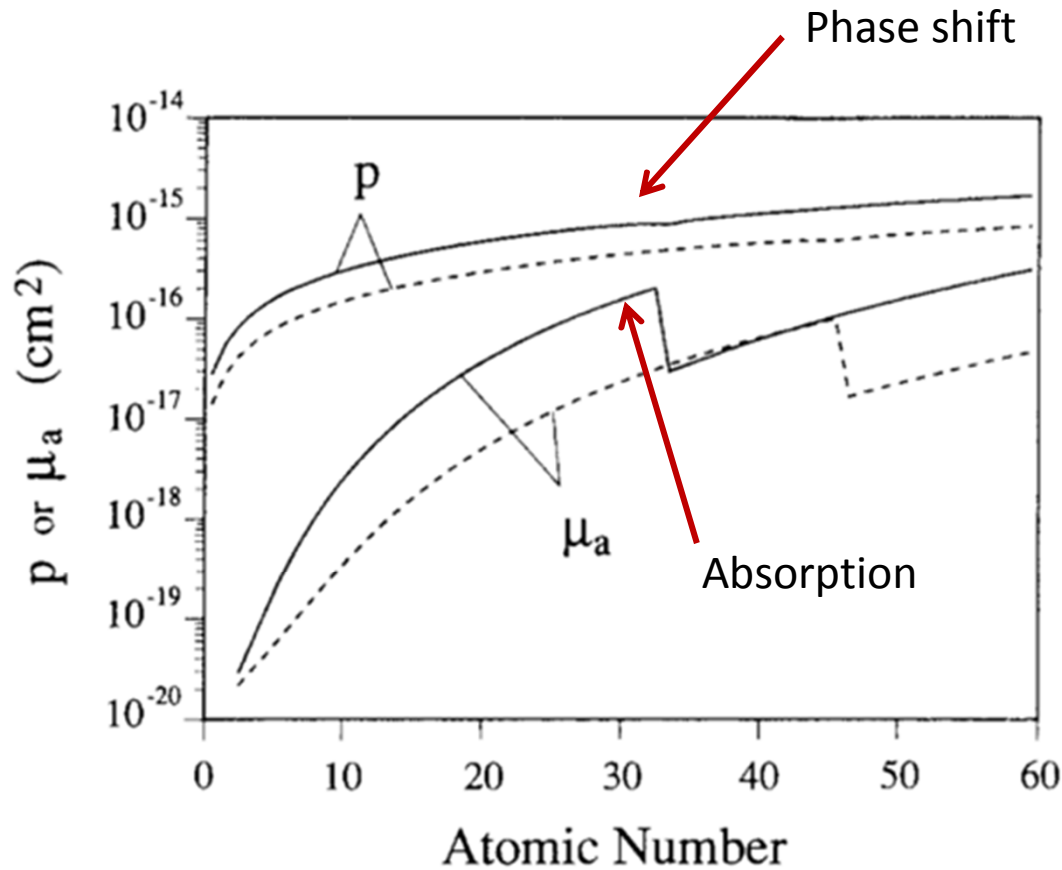
Dark-Field

Sharp contrast at boundaries.
Microstructures cause scattering.

To acquire only attenuation data leaves out significant information about the material properties

M. Bech, et. Al., Z. Med. Phys., **20**, 7, 2010.

Greater Sensitivity with Phase Contrast



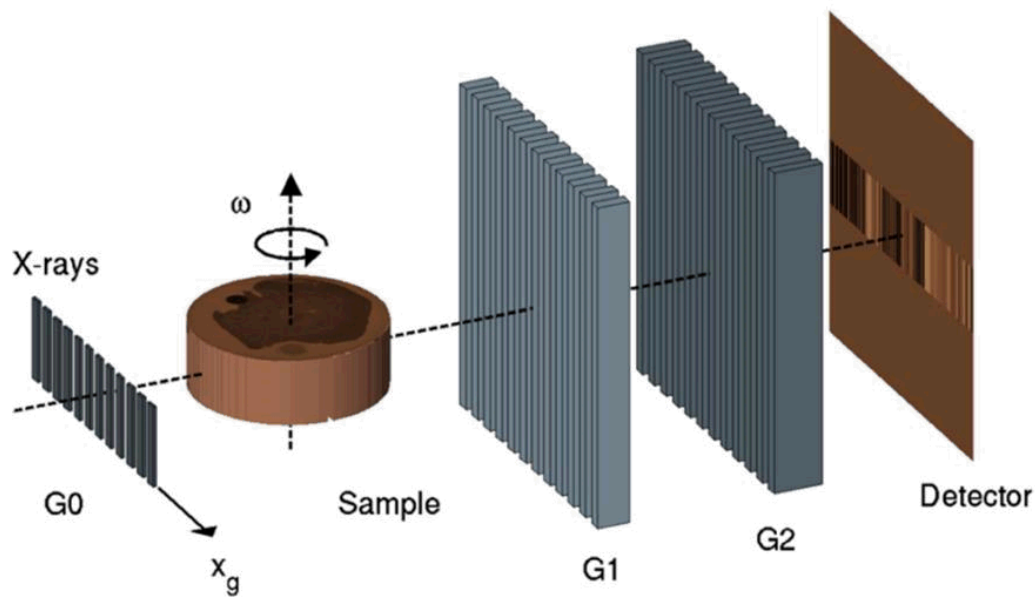
- X-ray absorption imaging is used for non-destructive imaging
- Poor sensitivity to low absorbing (low-Z) materials

Phase contrast
1000x more sensitive

FIG. 1. Atomic x-ray phase shift p and absorption μ_a for 1 Å (solid line) and 0.5 Å (dashed line) x-rays are plotted versus the atomic number Z . The value of p is almost a thousand times larger than μ_a for light elements.

Ref: A. Momose and J. Fukuda, Med. Phys., **22**, 375, 1995.

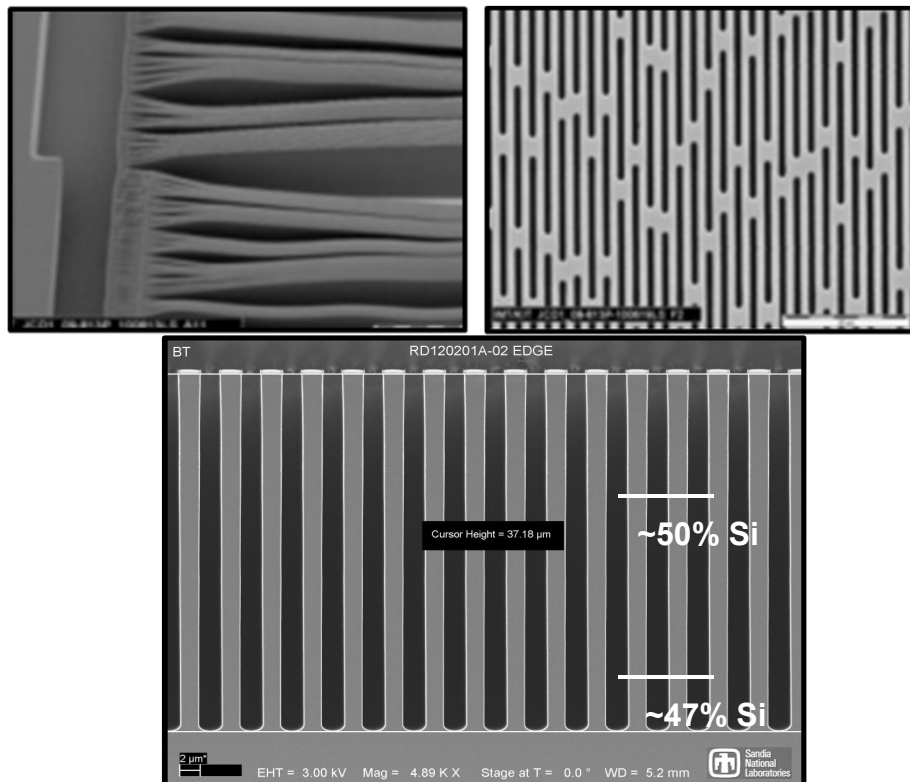
Talbot-Lau Interferometer



- Source grating: G0
 - Enables use of conventional x-ray tube
- Phase grating: G1
 - Imposes a modulated phase shift on wavefront
- Analyzer grating: G2
 - Converts narrow fringe pattern to intensity signal

**Source grating enables
lab-based XPCI**

- Unparalleled grating fabrication capability
- Advanced NDE/NDT facilities
- Materials science applications critical to national security



37μm deep

High Aspect Ratio 'Infinitely' Long Gold Gratings

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Electrochemical Deposition (ECD) offers unique capabilities for new device creation where traditional CMOS is unable to compare. Due to the nature of precise additive processing, as opposed to blanket material deposition, ECD is not hindered by line of sight constraints and keyhole effects inherent in traditional CMOS processes. Additionally, utilizing mold construction methods such as UV lithography, x-ray lithography, and Deep Reactive Ion Etching (DRIE), metal gratings can be realized at high aspect ratios for unique and complex device realization. In this work we've combined ECD with unique templates to realize high aspect ratio (as high as 50:1) gold gratings that up to 4 inches in length MetalMachining.sandia.gov

Metal Micromachining Team (MMT) Lithography Lab

- Hot Plate
- Wet Bench
- Resist Spinner
- Alignment Tool
- Vacuum Oven
- Microscopy

MMT Electroplating/Electroforming Lab

- Hard Gold
- Soft Gold
- Nickel Sulfate
- Copper Sulfate
- Electroless Nickel
- Custom Solutions

MMT Chemical Mechanical Planarization Lab

- Precision lapping, planarization and polishing capability
- Varying wheel composition to accommodate various material removal needs
- Controlled wafer, optics and device thinning
- Vacuum fixtures for 4 - 6 inch wafers
- Custom mounting fixtures to secure a multitude of devices

Enabling Technology for Phase Contrast Imaging

- Source Grating: Enables use of conventional x-ray source in the lab
- Phase Grating: Modulates the phase of the wavefront to form an interference pattern
- Analyzer Grating: Converts the interference pattern of the x-rays to an intensity signal that can be recorded with a detector

Benefits of XPCI

- No need for a synchrotron
- Higher resolution imaging than absorption based imaging
- Simplified optical arrangement
- Wide-Area Imaging

Greater than 25:1 Aspect Ratio Gratings Needed to Increase Absorption and Phase Contrast

- LIGA - resist swelling and structural instability
- CVD Tungsten - high stress (requiring parylene compensation) and non-conformal coatings
- DRIE Si can achieve > 50:1 aspect ratios and maintain high structural stability

Optimization of the Au Electroplating Process

- Aluminized 1 μm Thick Au Electroplating (Al Ti) of Pt for plating seed layer
- Tailored pulse plating for uniform deposition
- Multi-Pulse Control Plating for Au Electroplating
- Copper Seed Layer for Au Electroplating
- Full Wafer Gratings Plating


Au Source Gratings via UV Photoresist Trench Filling

- KMPR produces straight sidewalls with sufficient structural stability
- Conventional positive resists result in insufficient sidewall profiles
- Resist process is dependent on the "lifetime" of the wafer
- Lithography must be performed in a single day
- Plating must occur immediately after resist processing to prevent delamination and resist swelling

Realizing 16 in.² Grating Area

- Multiple electrical contacts
- Improved chemical flow
- Vibrating sample fixture
- Improved pulse plating regime

4 inch long 1 μm wide Au gratings 100,000:1 Length-Width Ratio!

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APPLICATION OF XPCI TO ADDITIVE MANUFACTURING

Porosity Measurements

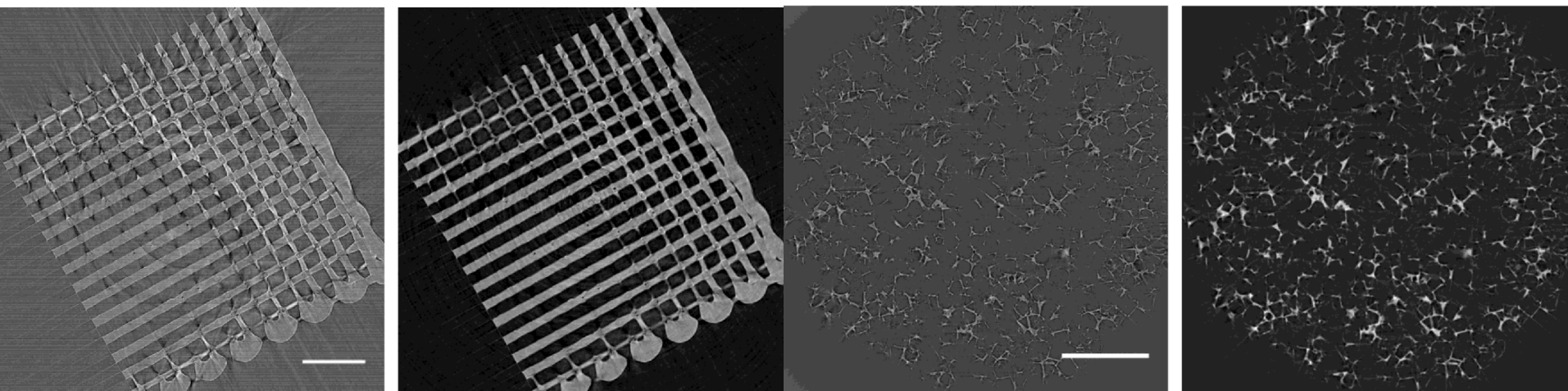
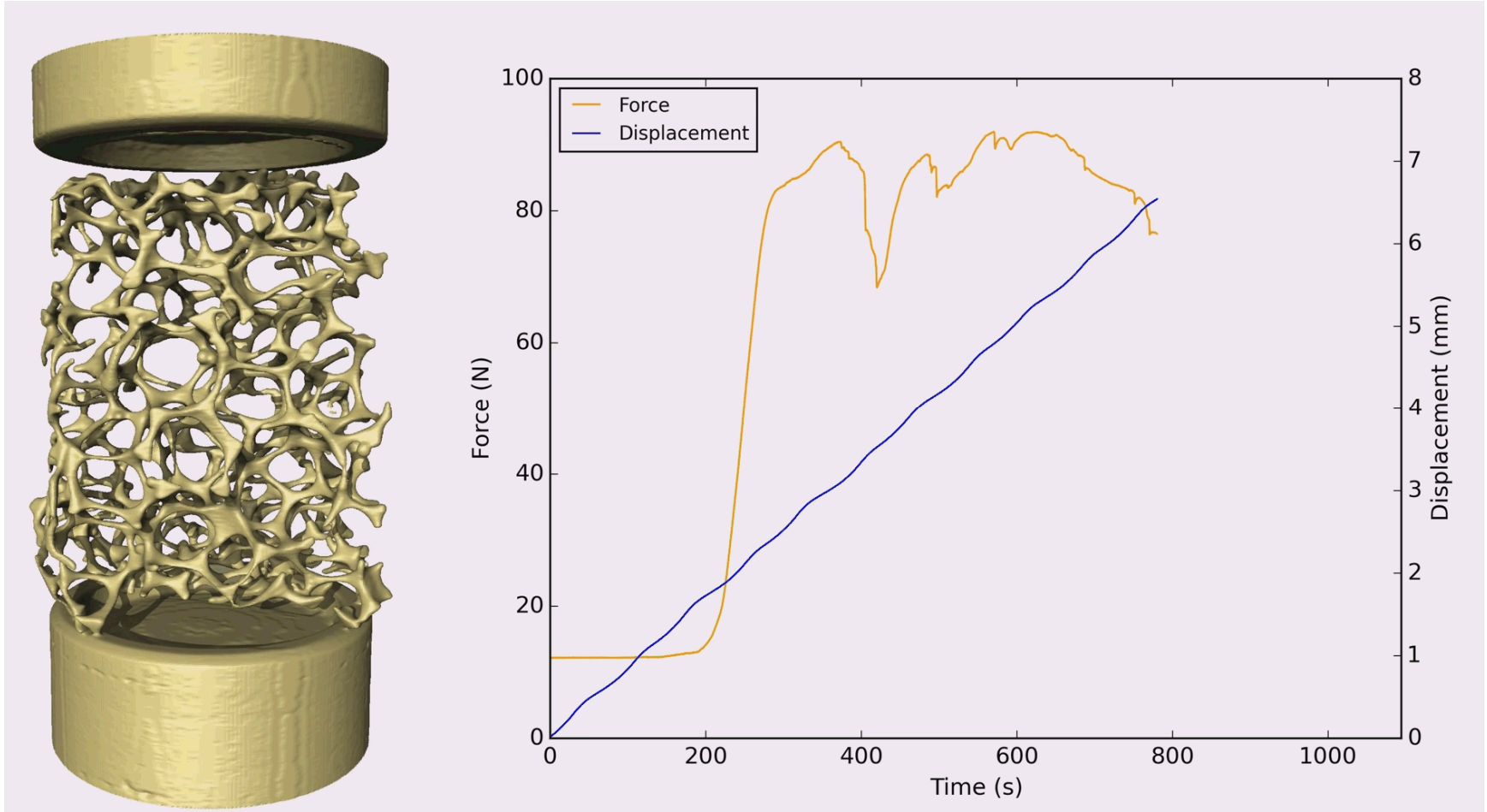


Table 1. Results of the quantitative image analysis of the considered VOIs.

	Additive Manufacturing	Conventional
Porosity [-]	47 %	89 %
Specific Surface Area [mm^{-1}]	4.87	7.45
Pore size: mean \pm SD (min \div max) [mm]	0.36 ± 0.02 ($0.27 \div 0.42$)	0.25 ± 0.08 ($0.03 \div 0.52$)
Throat size: mean \pm SD (min \div max) [mm]	0.23 ± 0.06 ($0.10 \div 0.37$)	0.12 ± 0.06 ($0.02 \div 0.36$)
Connection density [mm^{-3}]	4.20	22.61
Coordination number: mean \pm SD (max) [-]	3.8 ± 1.1 (9)	5.0 ± 3.5 (34)

Ref: Brun, F., et. al. (2013). *Journal of Instrumentation*, 8, 1–6.

Quantitative Analysis



Ref: XRE Engineering, Van Loo Lab, Ghent, Belgium

XPCI to Validate & Verify 3D Printed Parts

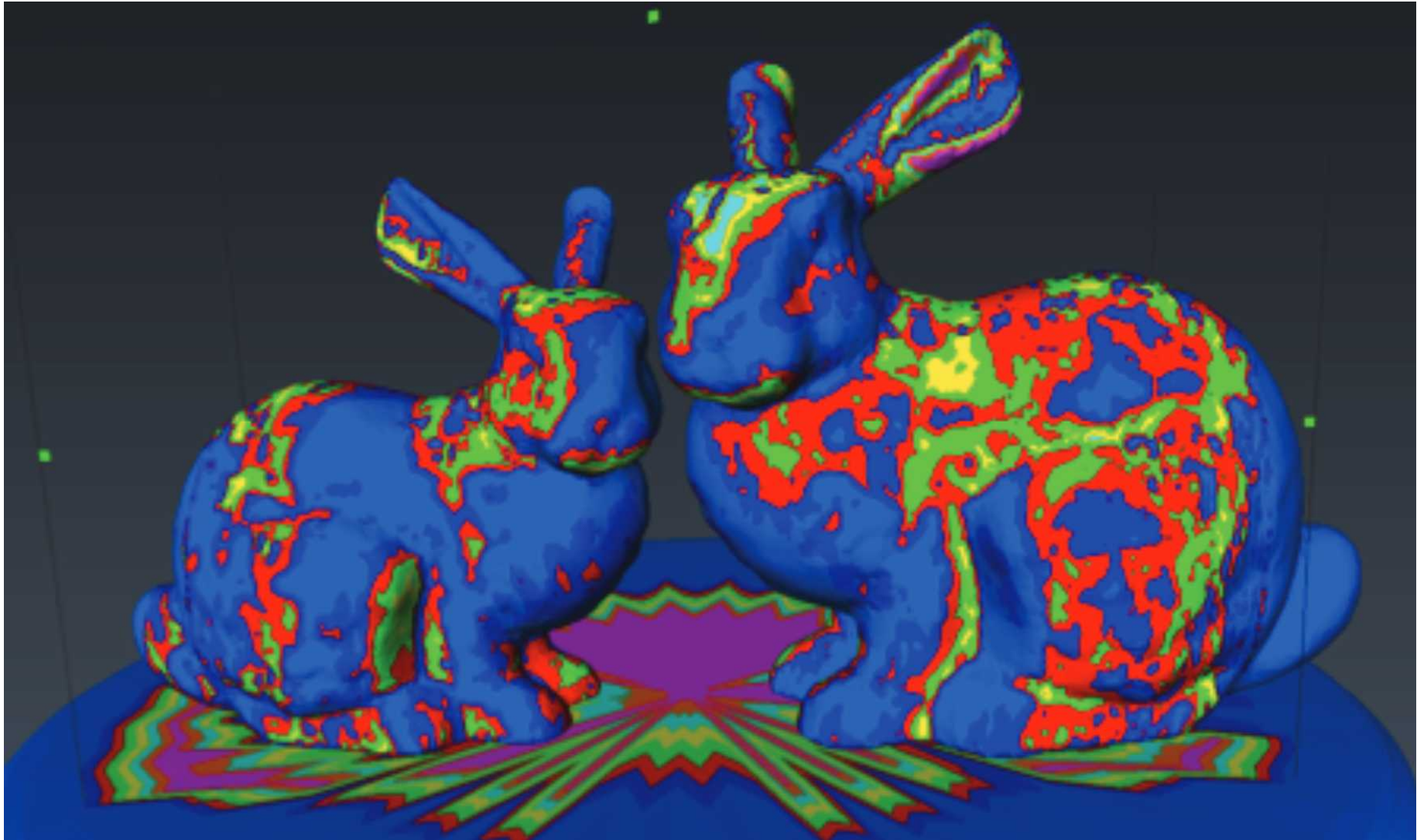
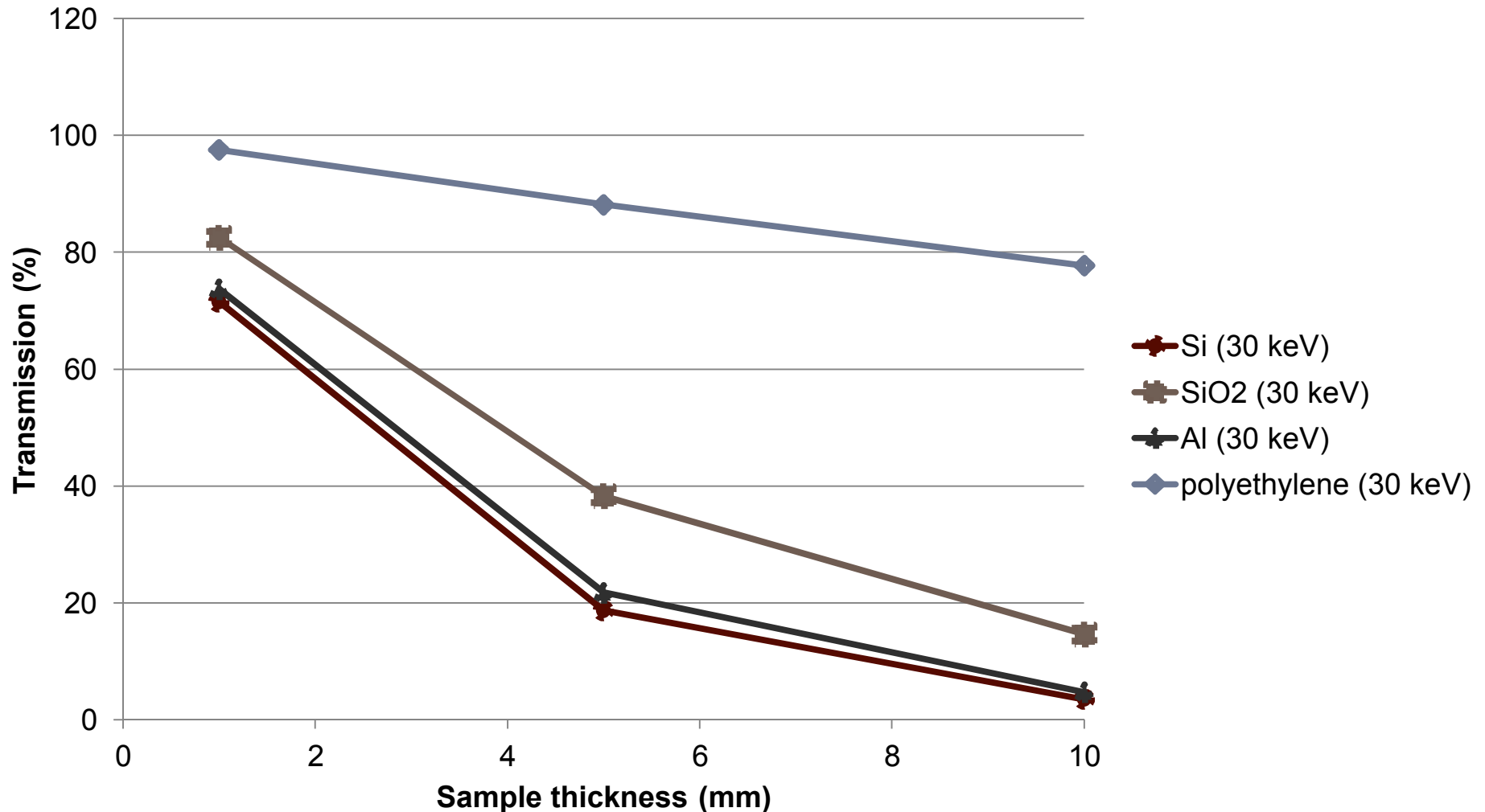


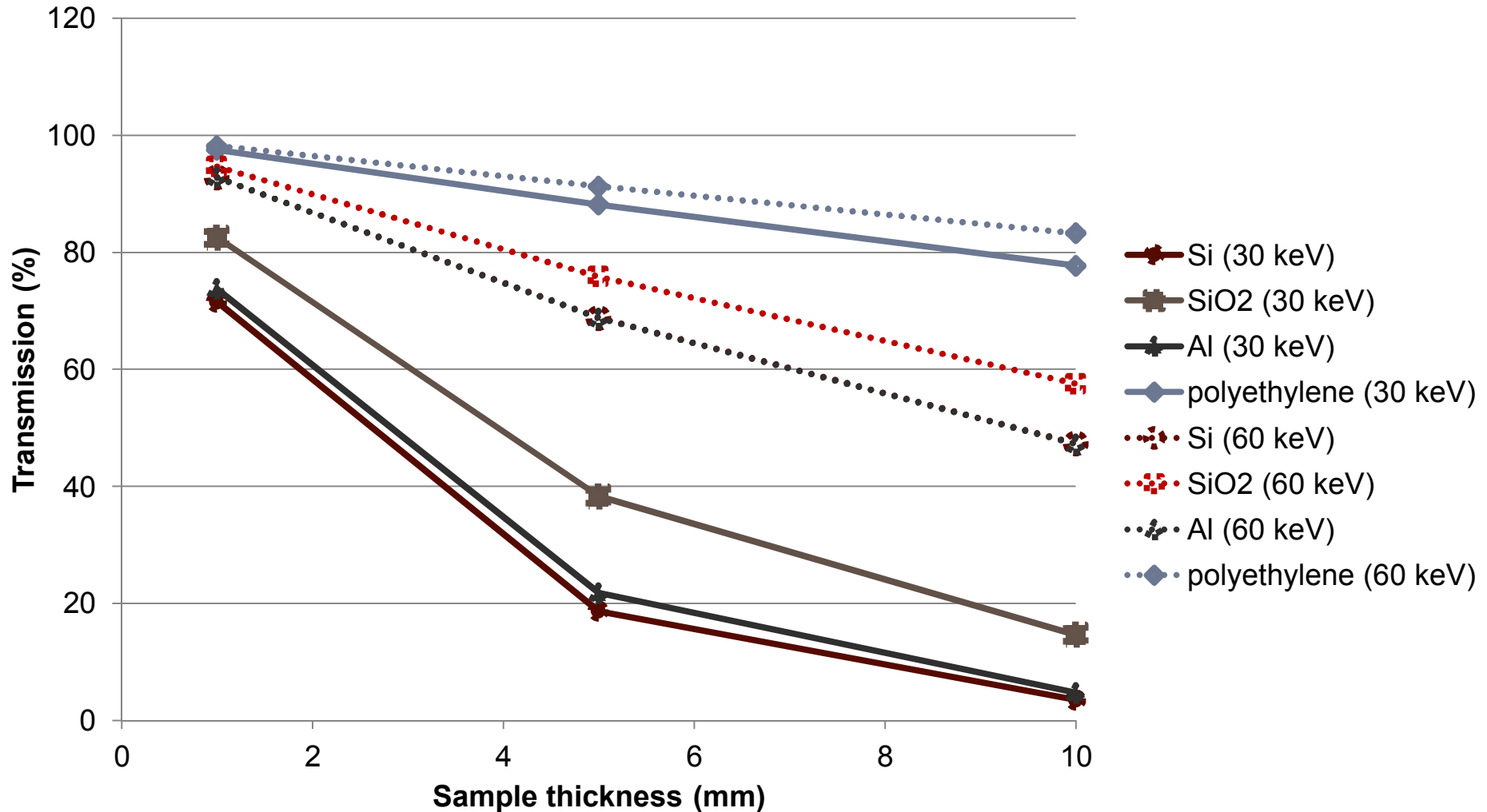
Figure from Butler lab, LSU-CAMD

X-Ray Transmission as a function of sample thickness

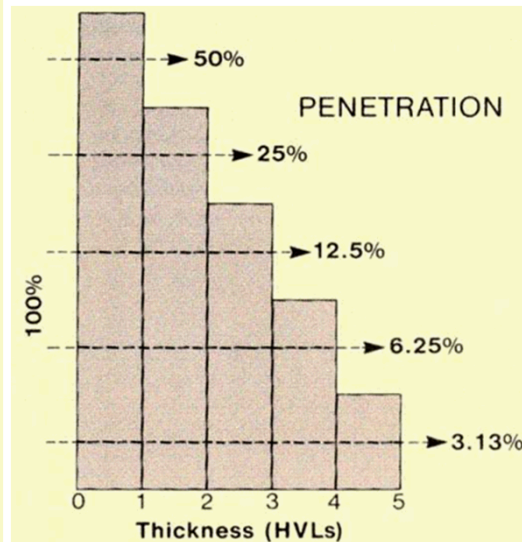
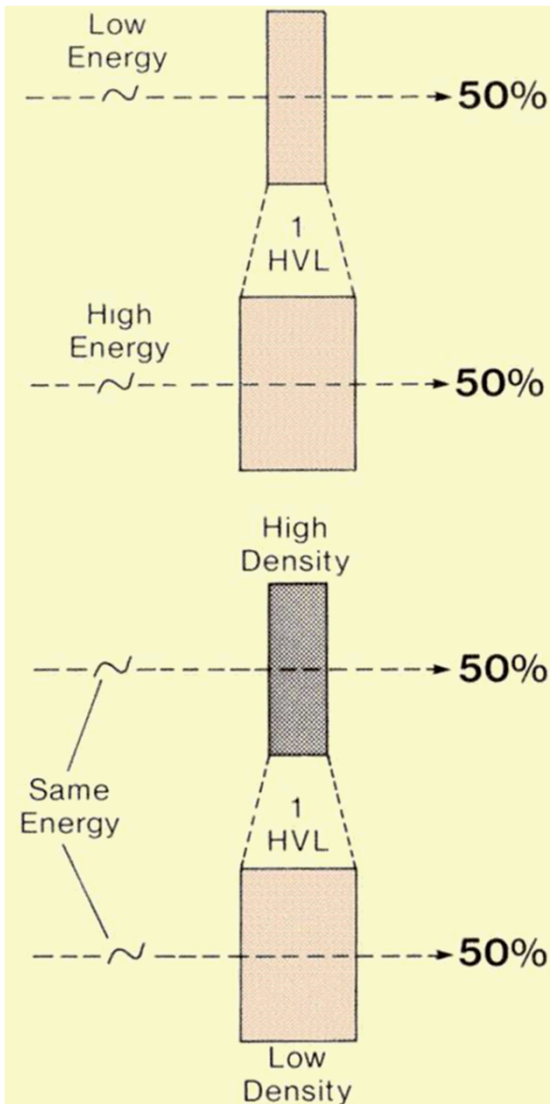


Effect of Increasing X-ray Energy

X-Ray Transmission as a function of sample thickness



Representative Half Value Layers



$$I = I_0 e^{-\mu x}$$

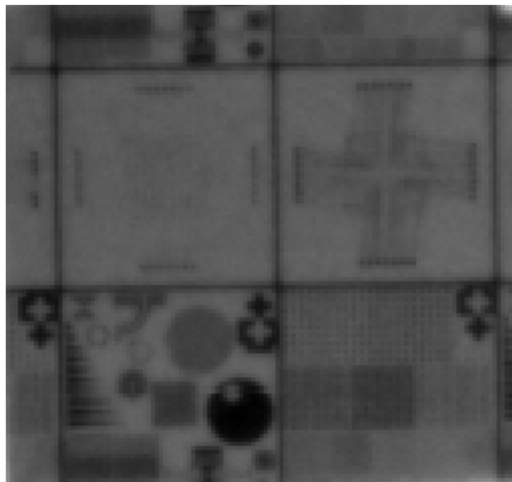
$$0.5 = 1.0 e^{-\mu x}$$

$$HVL = \frac{0.693}{\mu}$$

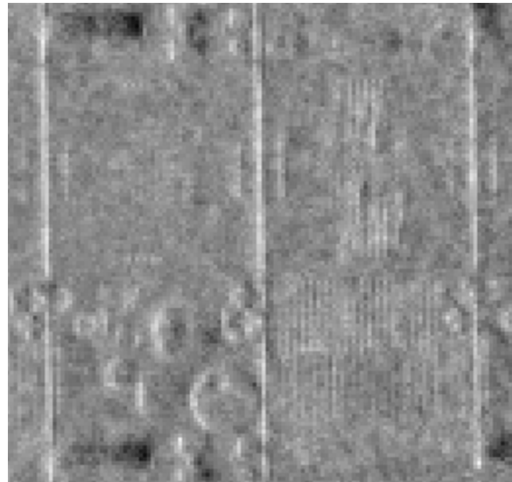
	HVL (mm)	
	30 keV	60 keV
Polyethylene	27.54	37.83
Water	18.45	33.66
Mylar	16.69	26.88
Teflon	7.65	16.38
Borosilicate	3.89	12.86
Concrete	3.14	11.33
Bone	2.71	11.47
Aluminum	2.28	9.24
Silicon	2.07	9.27
304L steel	0.11	0.73
CdTe	0.05	0.17
Lead Glass	0.05	0.29

HVL cartoons from: <http://www.sprawls.org/ppmi2/RADPEN/>

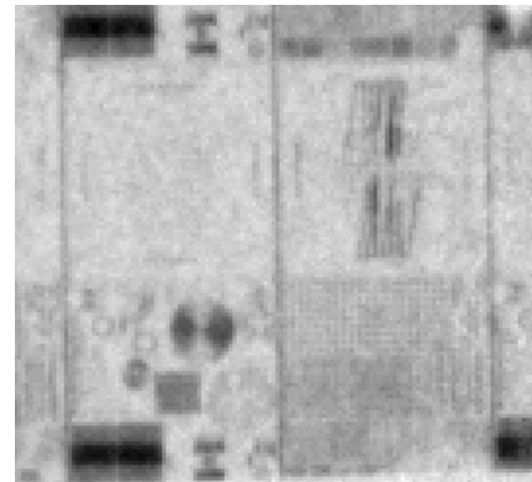
- Critical to assuring the functionality and security of integrated circuits (ICs)
 - “Slice and dice” inspection
 - Randomly selected samples
 - Labor intensive
 - Limited imaging depth
 - Complicated sample preparation
 - Limited in materials that can be inspected
- XPCI: non-destructive imaging of internal composition
 - 1000x more sensitive than x-ray absorption imaging for low-Z materials
 - Differences between ICs
 - Cracks or intentional/unintentional addition and deletion of materials



Absorption



Phase Contrast

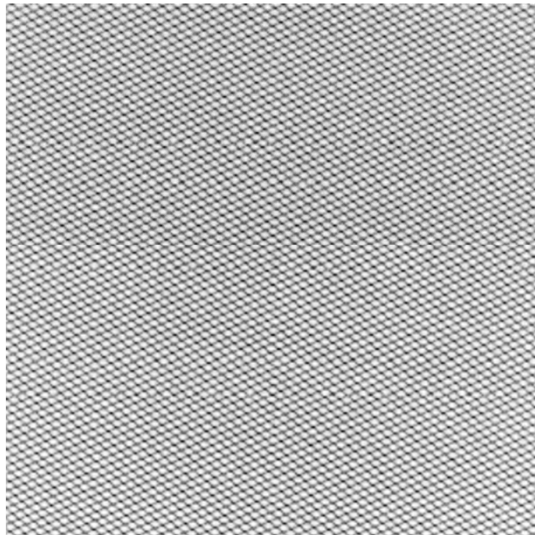


Dark Field

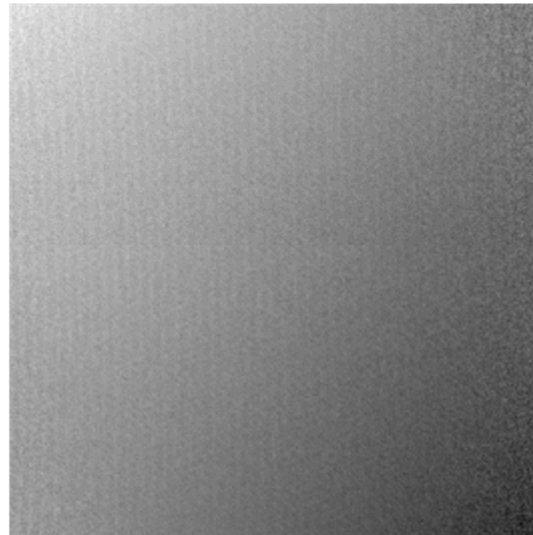
Grafoil in Aircraft Laminate

8 Ply laminate, copper mesh side, single 8-step sequence GF

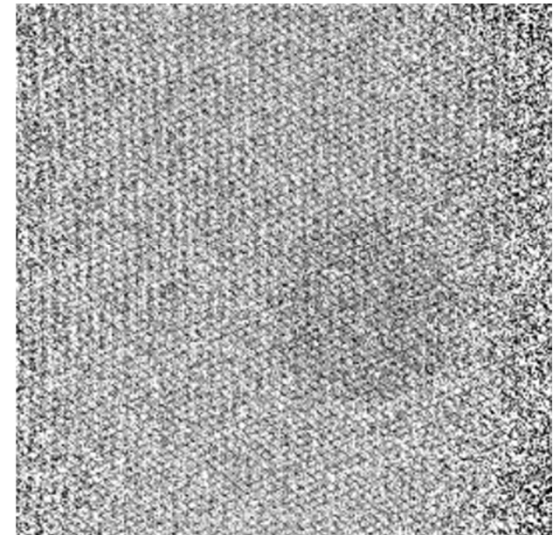
Transmission



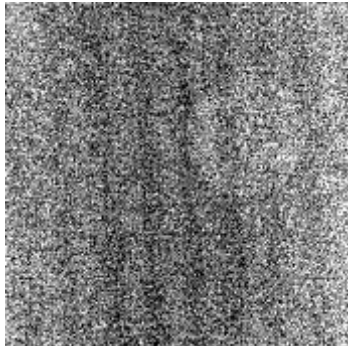
Differential Phase



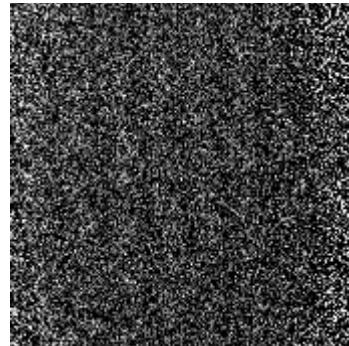
Dark Field



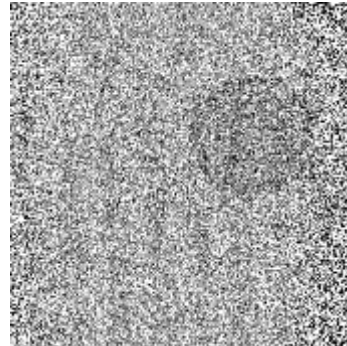
Transmission



Differential Phase



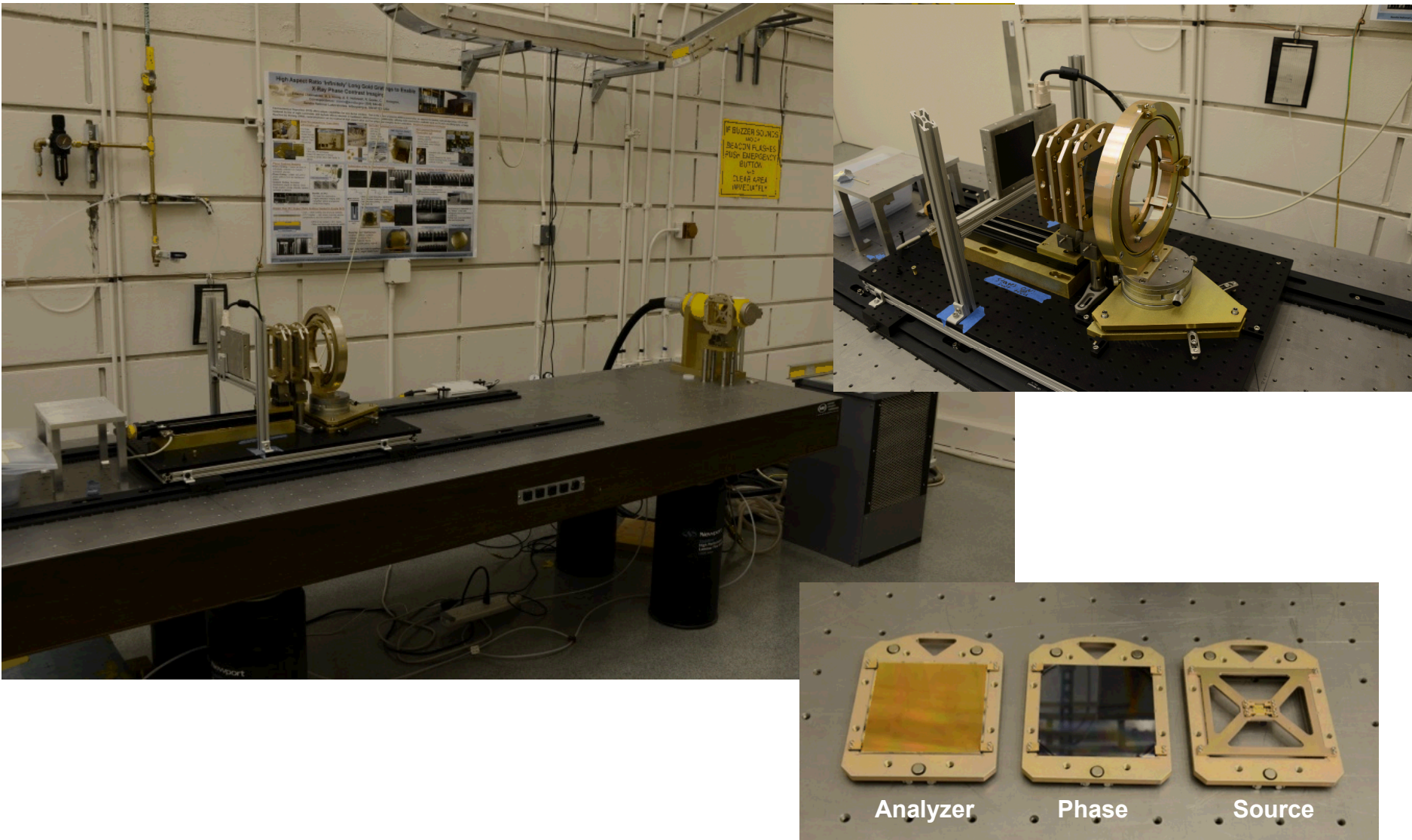
Dark Field



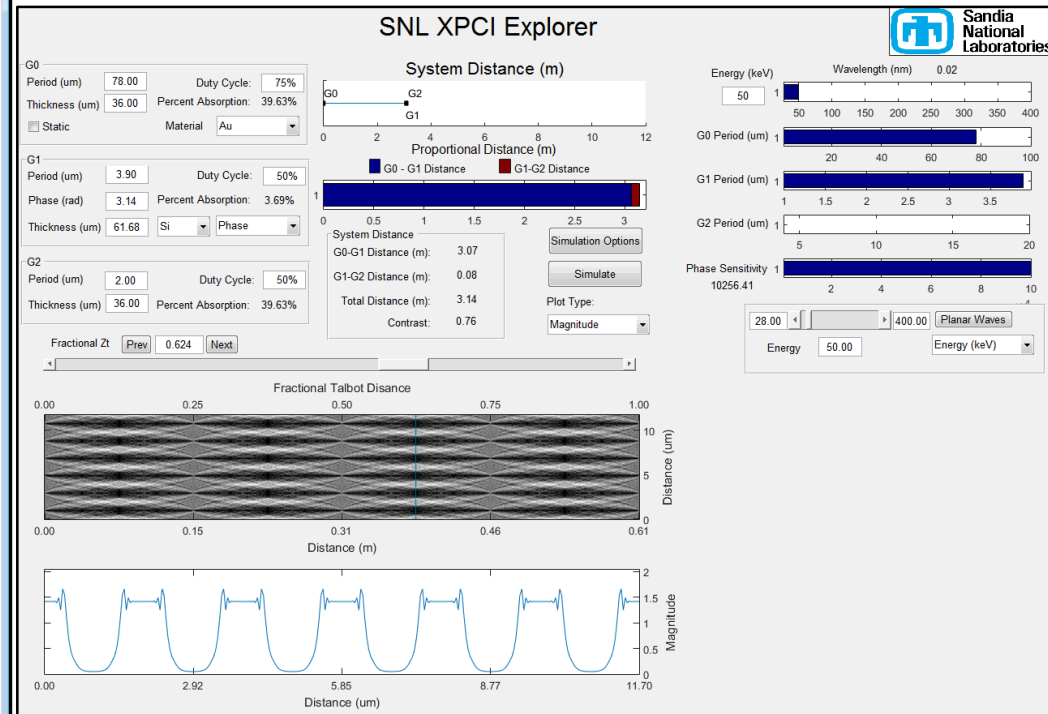
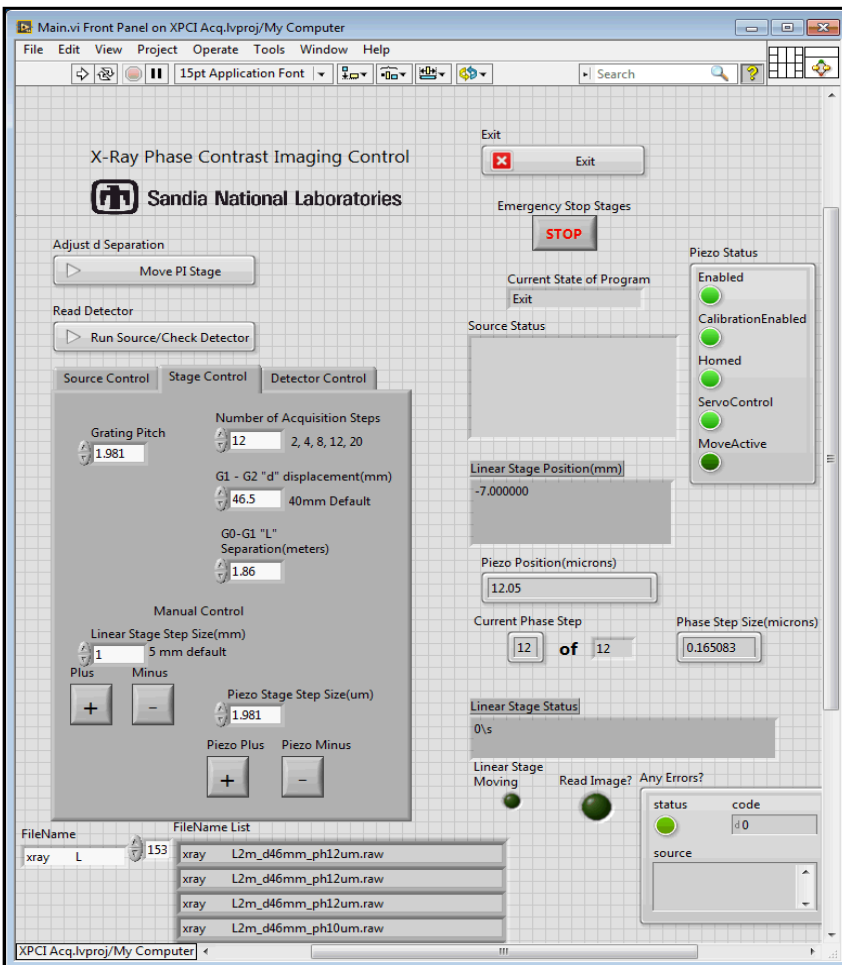
8 Ply laminate
NO copper mesh side
single 8-step sequence

STATUS AT SANDIA-NM

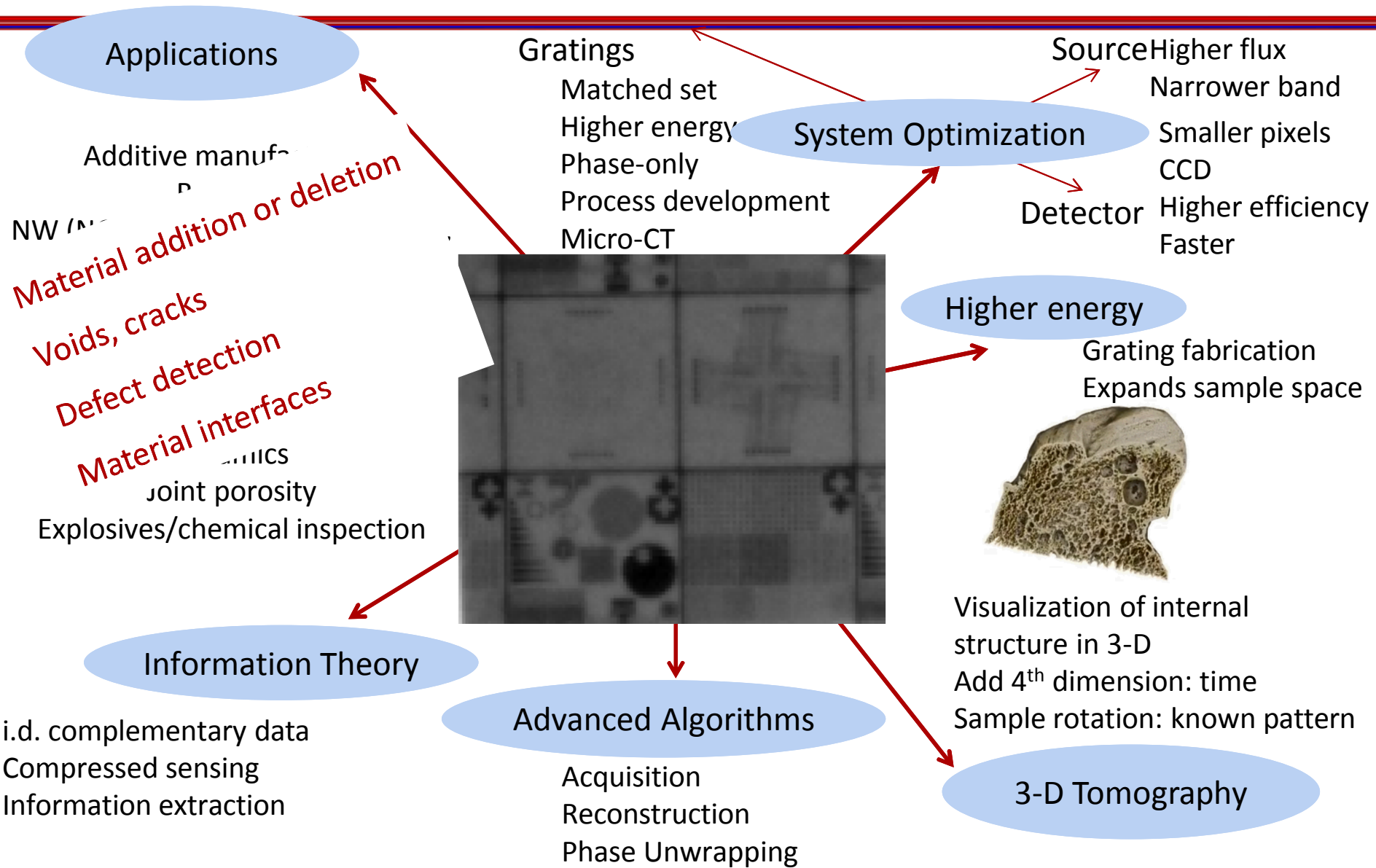
Laboratory Based System at Sandia



Interface & Modeling



Growth Opportunities



Acknowledgements



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Project Manager: Brad Gabel

Funding: Laboratory Directed Research and Development (LDRD)

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