

# Preliminary Residual Stress Mapping of GRCop-84 Fabricated by SLM

**Robert Minneci**

**The University of Tennessee**

Claudia Rawn (UTK), Jeff Bunn (ORNL), Jared Floyd (UTK), Zachary  
Jones (NASA)



# Material Overview: GRCop-84



## Glenn Research Copper Alloy

NASA Glenn Research Center

- Starting At%: Cu-8 Cr-4 Nb

- Copper Matrix:

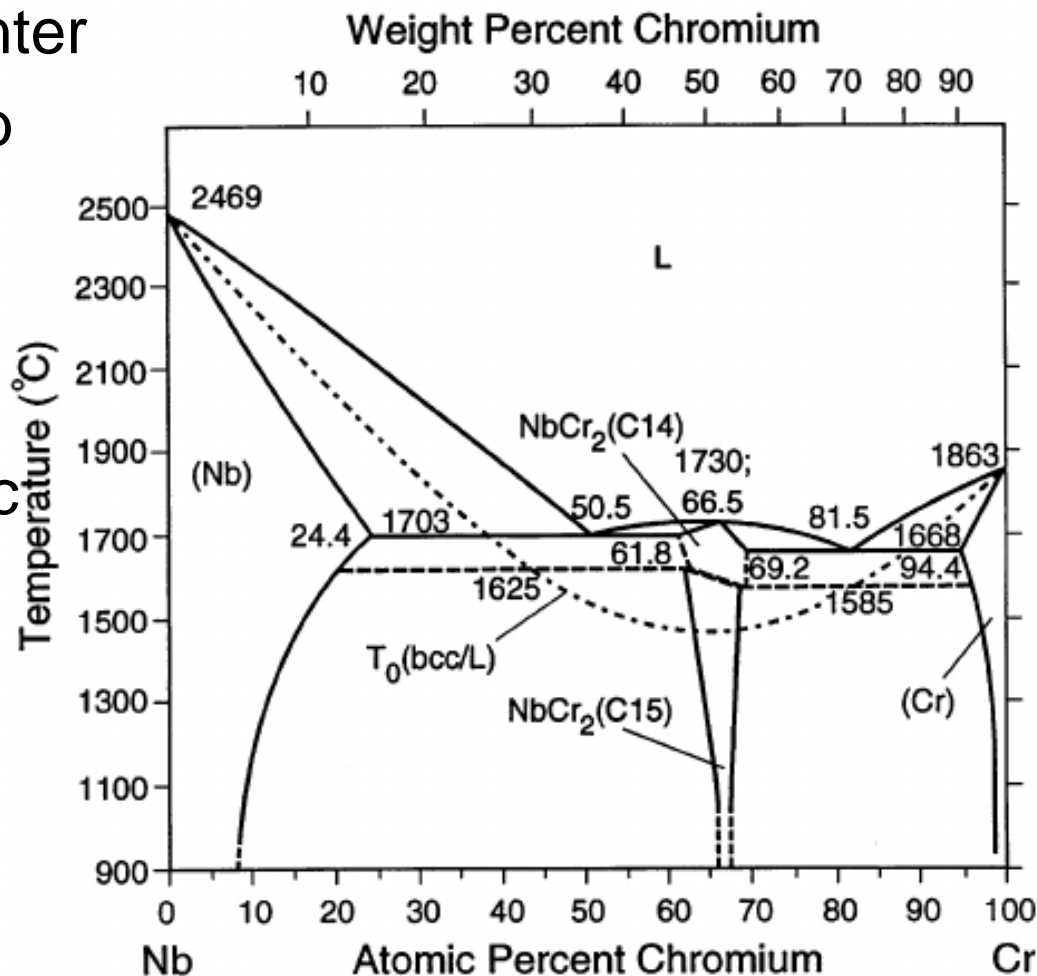
fine intermetallic

dispersion  $\text{Cr}_2\text{Nb}$

Laves phase C15 - cubic

Developed for reusable launch vehicles

- Competing with other copper precipitate-strengthened alloys
  - NARloy-Z, AMZIRC, Oxygen Free Cu (OFHC Cu)



Phase diagram from: Thoma et. al. *Elastic and Mechanical Properties of Nb(Cr,V)<sub>2</sub> C15 Laves phases*



# SpaceX makes aerospace history with successful launch and landing of a used rocket

by Loren Grush | @lorengrush | Mar 30, 2017, 7:07pm EDT

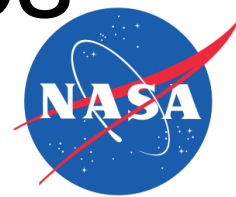
f SHARE | TWEET | in LINKEDIN



After more than two years of landing its rockets after launch, SpaceX finally sent one of its used Falcon 9s back into space. The rocket took off from Cape Canaveral, Florida, this evening, sending a communications satellite into orbit, and then landed on one of SpaceX's drone ships floating in the Atlantic Ocean. It was round two for this particular rocket, which

<https://www.theverge.com/2017/3/30/15117096/spacex-launch-reusable-rocket-success-falcon-9-landing>





# Application Related Properties

## High Heat Flux Applications $\leq 700$ °C

Excellent high temperature properties

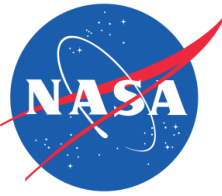
- Low thermal expansion
  - 7% less than pure Cu at hot wall (400-600°C)
  - Significantly higher lifetime
- High thermal conductivity
  - 305-320 W/m\*K (75-84% of pure Cu)
- High electrical conductivity
- Tensile strength
  - Excellent retention at high temp.
- High creep resistance
- Long low-cycle fatigue lifetime
- Enhanced oxidation resistance



Hot-fire test of GRCop-84 Liner, image from:  
Loewenthal, William & Ellis, David. *Fabrication of GRCop-84 Rocket Thrust Chambers*. Slide 16



# Microstructure



- $\text{Cr}_2\text{Nb}$  has minimal solid solubility with copper
  - Minimizes coarsening, grain size remains consistent
    - 1-5  $\mu\text{m}$  copper matrix grains
  - Extremely stable up to 800°C
- $\text{Cr}_2\text{Nb}$  particles pin copper grain boundaries
  - **Hall-Petch** mechanism
    - Dependence of strength on grain size (2/3)
  - **Orowan** strengthening
    - Dislocation bow from particles (1/3)
- Forms layer of Cr-Nb oxides below copper oxide layer
  - Slight excess of Cr to prevent Nb-H embrittlement





# Additive vs Traditional

AM outperforms traditional

- **Geometry**
  - Thinner wall capabilities
    - Better thermo-mechanical properties
  - Built-in cooling channels
    - Increased temperature margins
- **Fabrication time**
  - 3-9 months to 1 month lead
    - Compared to HIPed and wrought
  - Can be built in-house (MSFC)
- **Properties (after HIP)**
  - Porosity reduction
    - Greater or equal to traditional
    - Fully dense (greater than 99.9%)

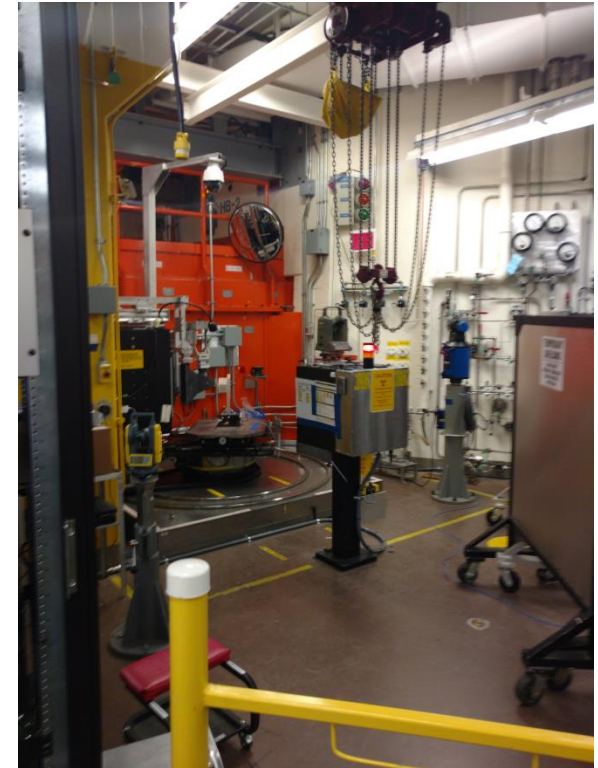


Image credit: NASA/MSFC/Emmet Given *Nasa 3-D Prints First Full Scale Copper Rocket Engine Part*



# Neutrons and GRCop-84

- Few existing neutron studies
  - Effectively none for AM GRCop-84
  - Why?
    - AM development
- Still under development
  - Needs supporting data
  - Direct benefit of results
  - Carries general AM concerns
- Proposal Goals
  - Initial simplicity for familiarization
    - Begin with bulk residual stress/strain
    - Thermal misfit
  - Conduct multiple studies building up to comprehensive study
  - Potential modeling



# Residual Stress

- High thermal gradients
  - $10^4$  to  $10^6$  K/s cooling
  - Excess RS can lead to plastic deformation
    - Before heat treatment can be performed
    - Build has to be restarted, significant loss



Image credit: NASA/MSFC/Emmet Given *Nasa 3-D Prints First Full Scale Copper Rocket Engine Part*



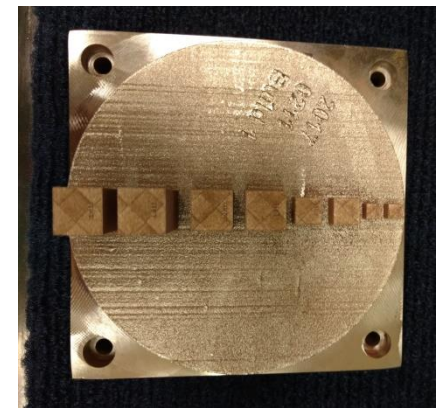
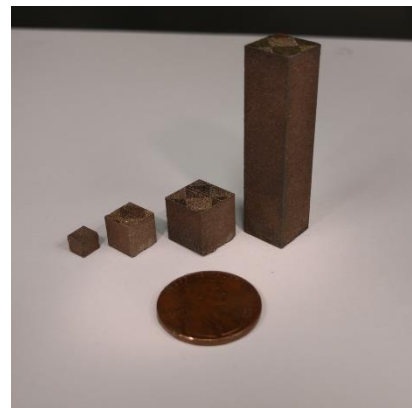
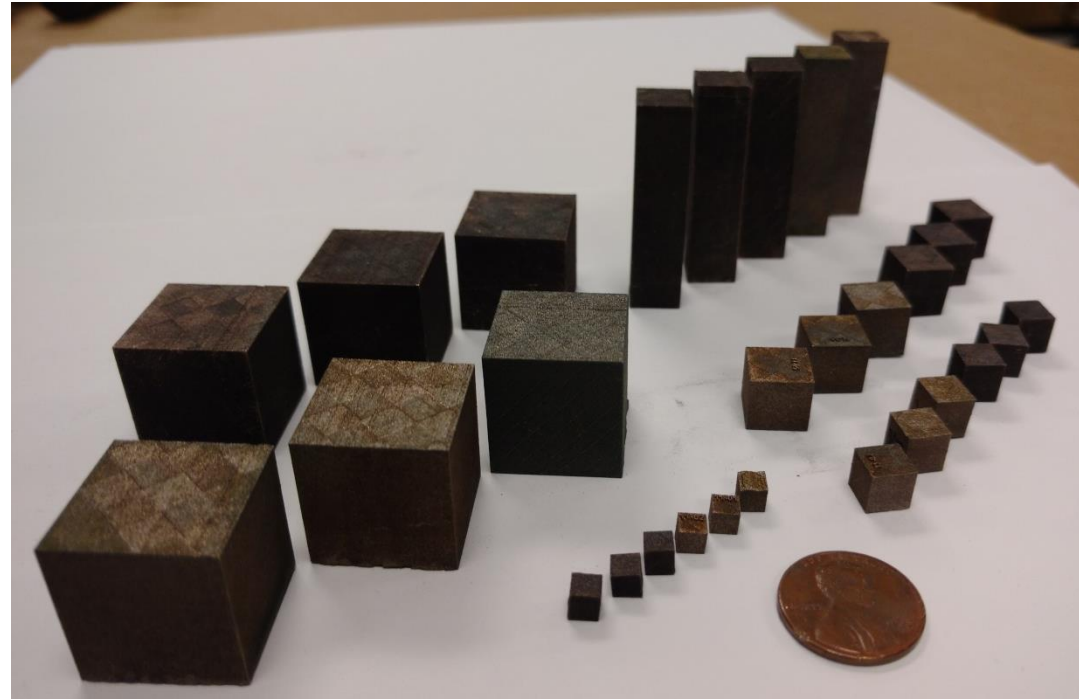
# Samples - MSFC

Built on Concept Laser M2

- SLM – Chess Pattern
- Powder bed fusion
- Pre- and post-HIP
- Removed from build plate by EBM

Simple geometries

- 20,10, 7, and 4 mm cubes
- 10x10x40 pillars



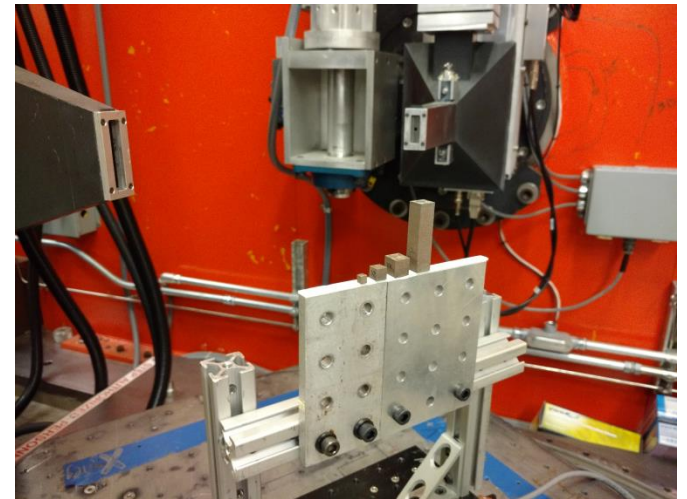
# GRCop-84 Beamline Setup

Experiment conducted at HFIR

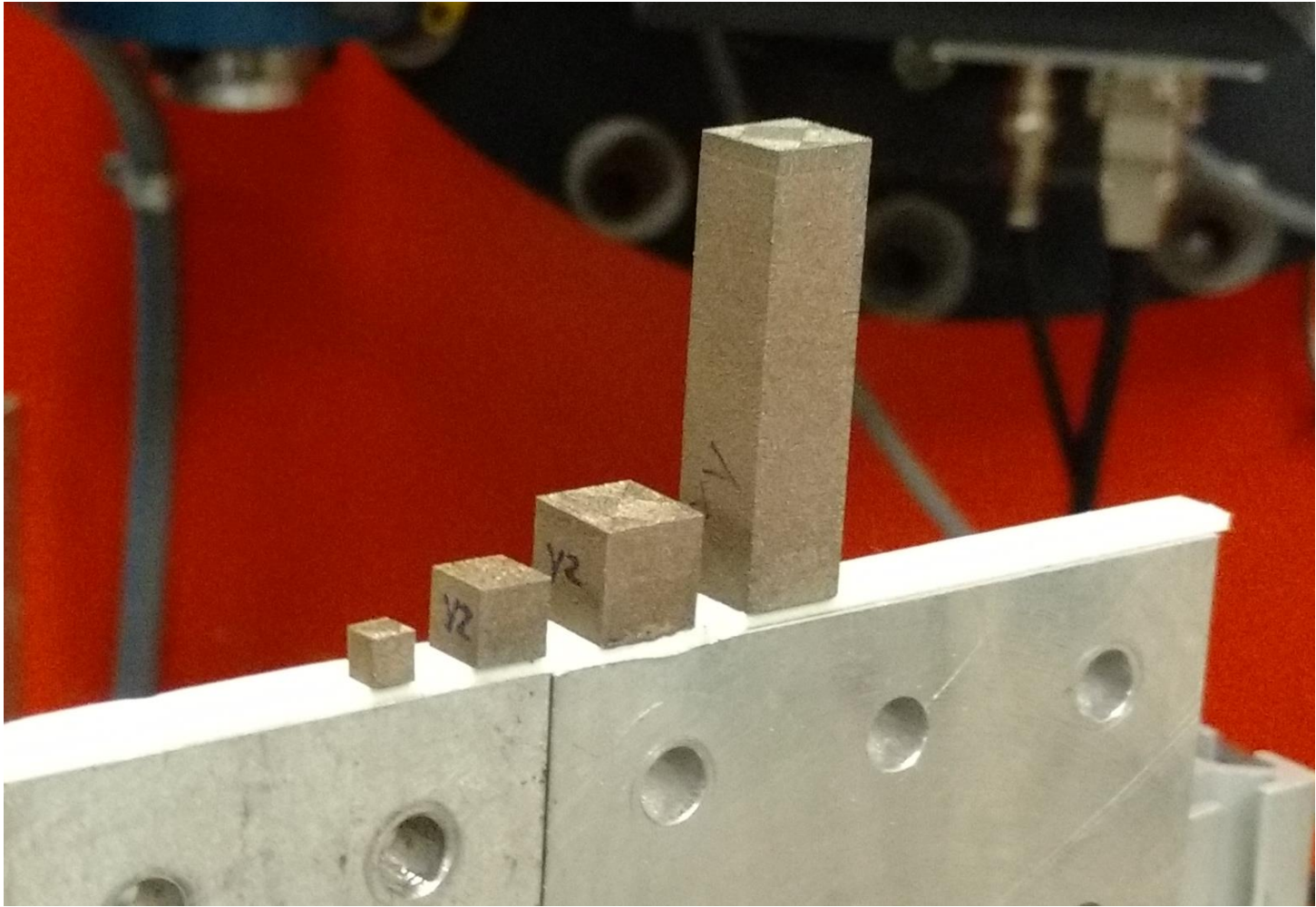
- NRSF2 HB2B
  - Neutron Residual Stress Mapping Facility
- May 2017

Scanning Parameters

- 2 mm<sup>3</sup> gauge volume
  - <5 minutes per point
    - 40 minutes for 1 mm<sup>3</sup> gauge vol.
- 422 monochromator  $\sim 1.54 \text{ \AA}$ 
  - Cu 311 reflection  $\sim 90^\circ 2\theta$



# Samples Measured



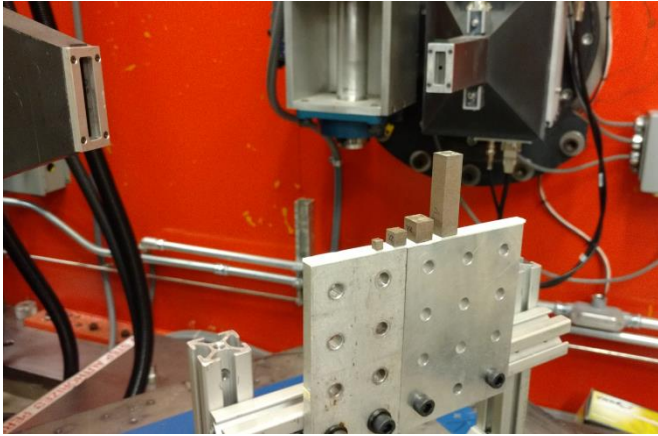
- 4, 7, and 10 mm cube
- 10 x10 x 40 mm pillar



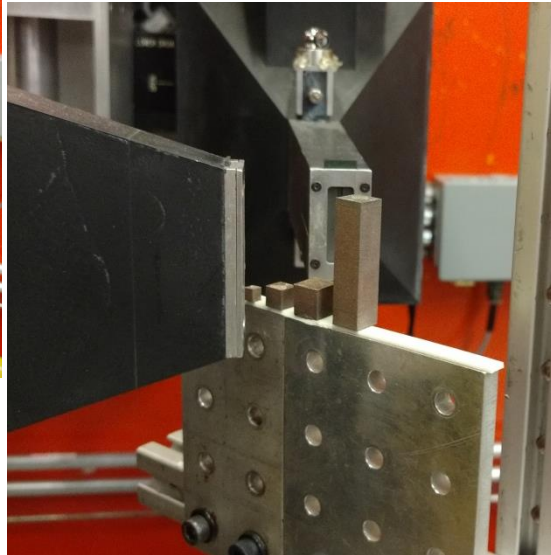


# Sample Orientations/Mounting

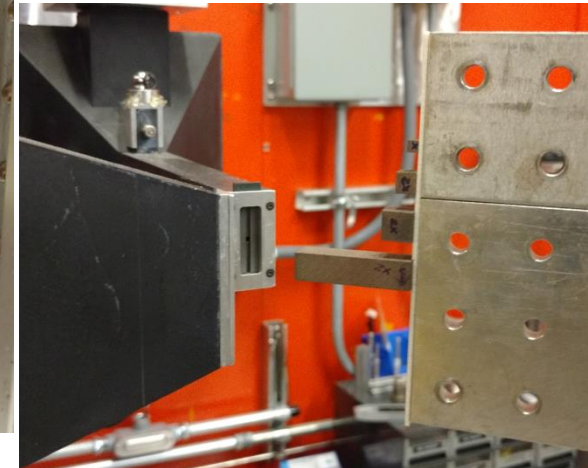
X-Direction



Y-Direction



Z-Direction



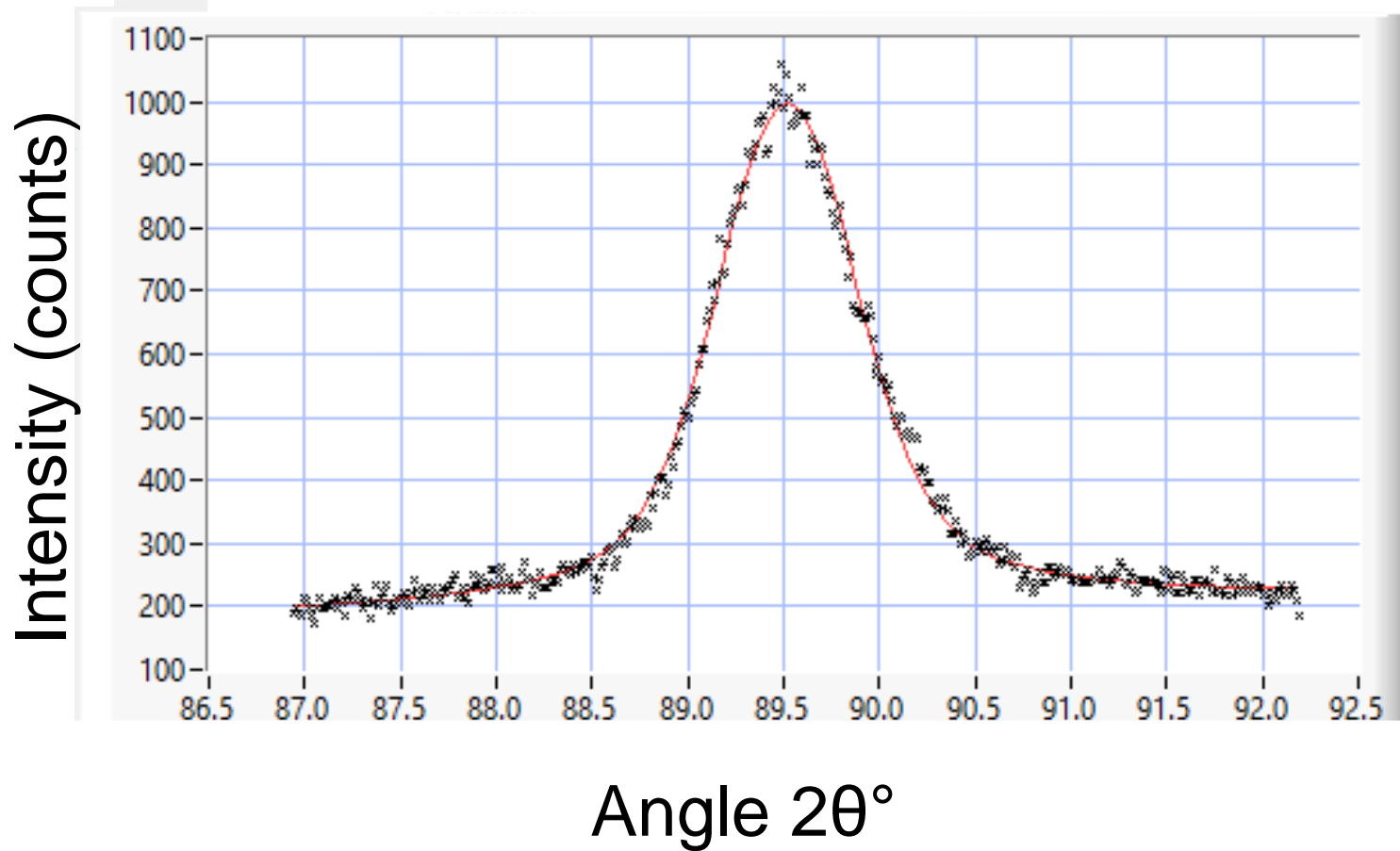
Strain Calculation

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0}$$

Stress Calculation

$$\sigma_{ij} = \frac{E}{1 + \nu} \left( \varepsilon_{ij} + \frac{\nu}{1 - 2\nu} (\varepsilon_{11}^{hkl} + \varepsilon_{22}^{hkl} + \varepsilon_{33}^{hkl}) \right)$$

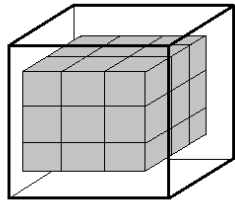
# Residual Strain Measurement



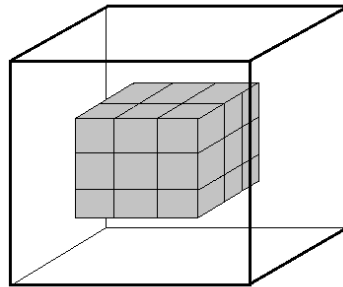


# Beamline Experiment

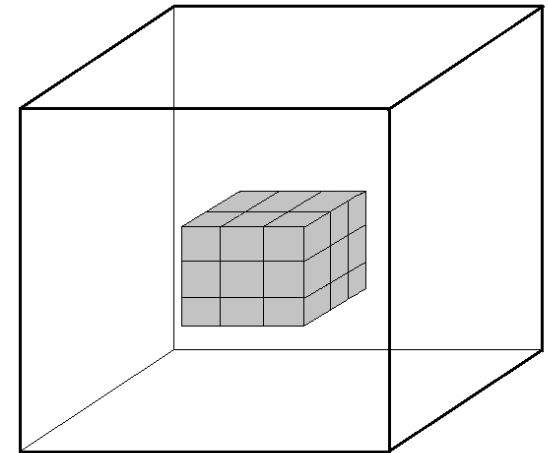
## 1. Volumetric Study



4 mm

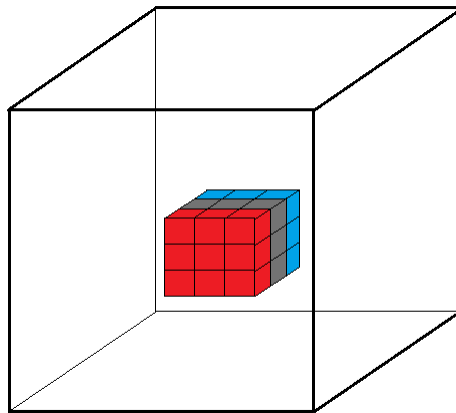


7 mm

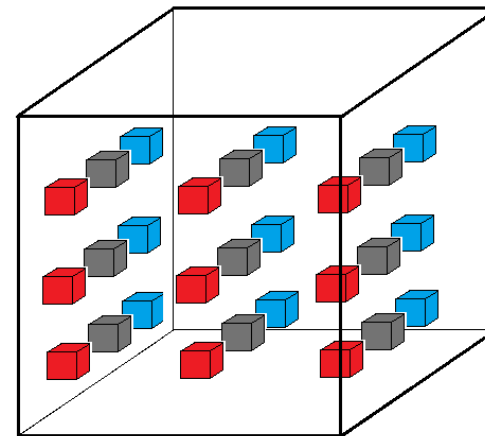


10 mm

(only compact)



Compact

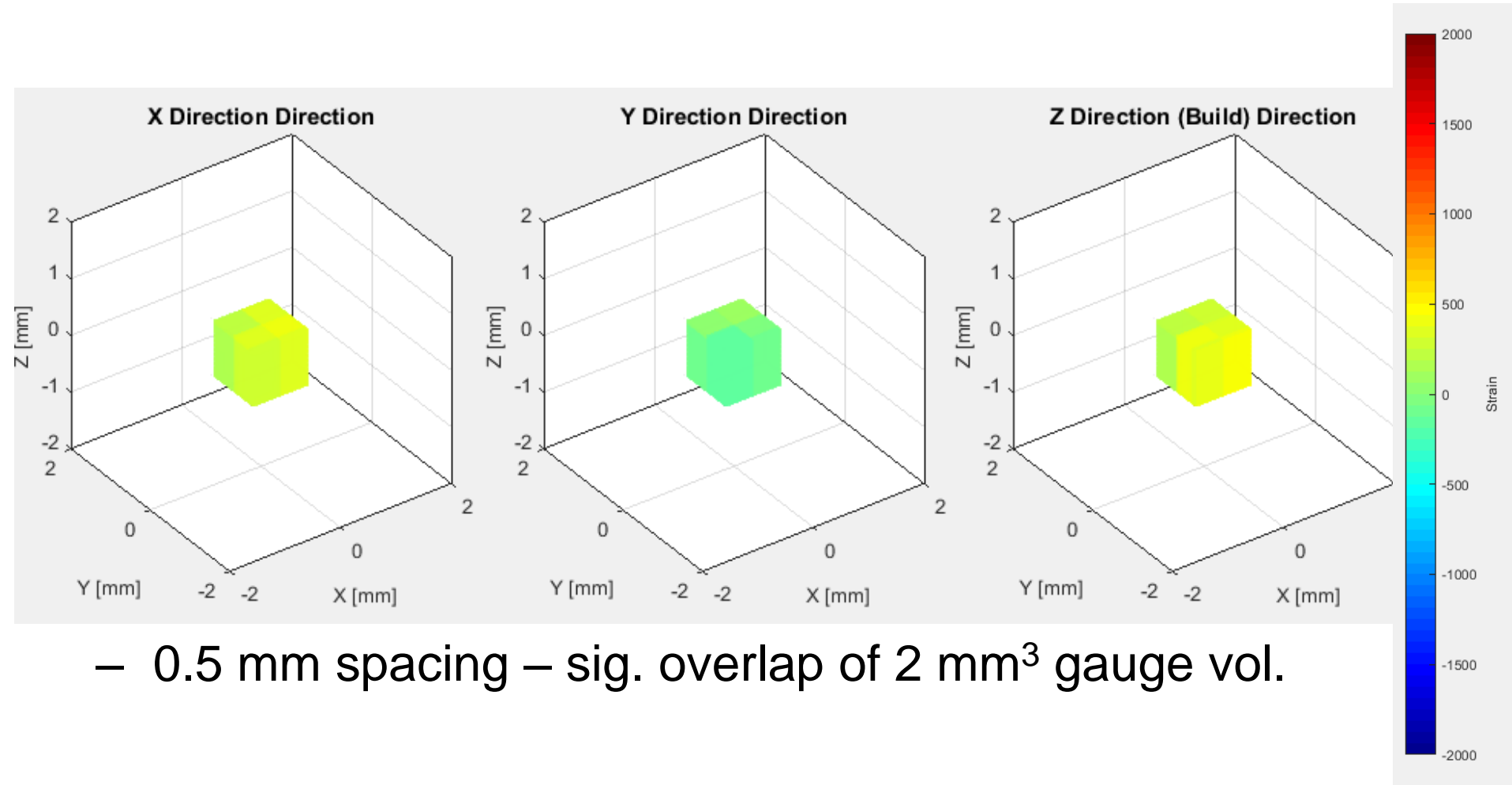


Spread



# Volumetric Results

## 4 mm cube strain



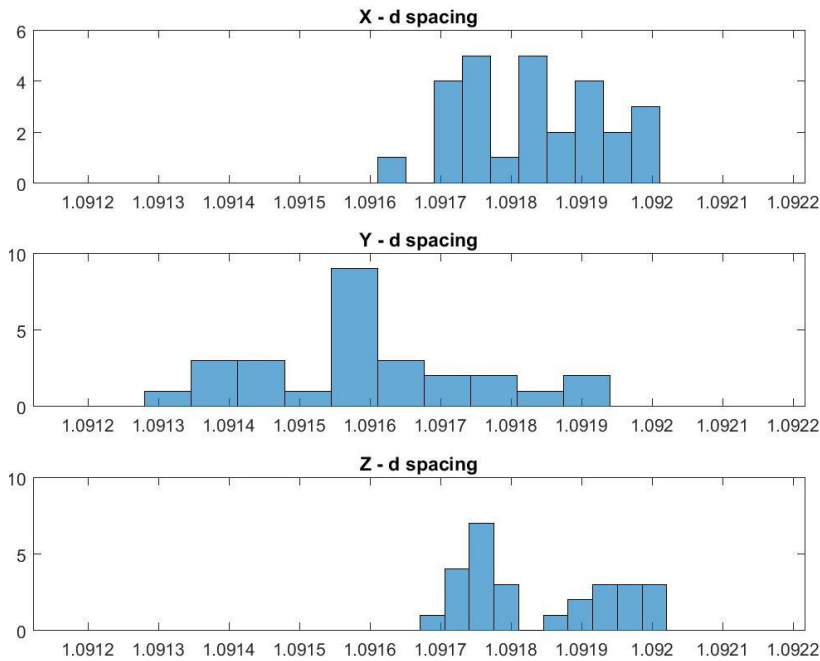
– 0.5 mm spacing – sig. overlap of 2 mm<sup>3</sup> gauge vol.

- Figures generated using Matlab code created by Jeff Bunn

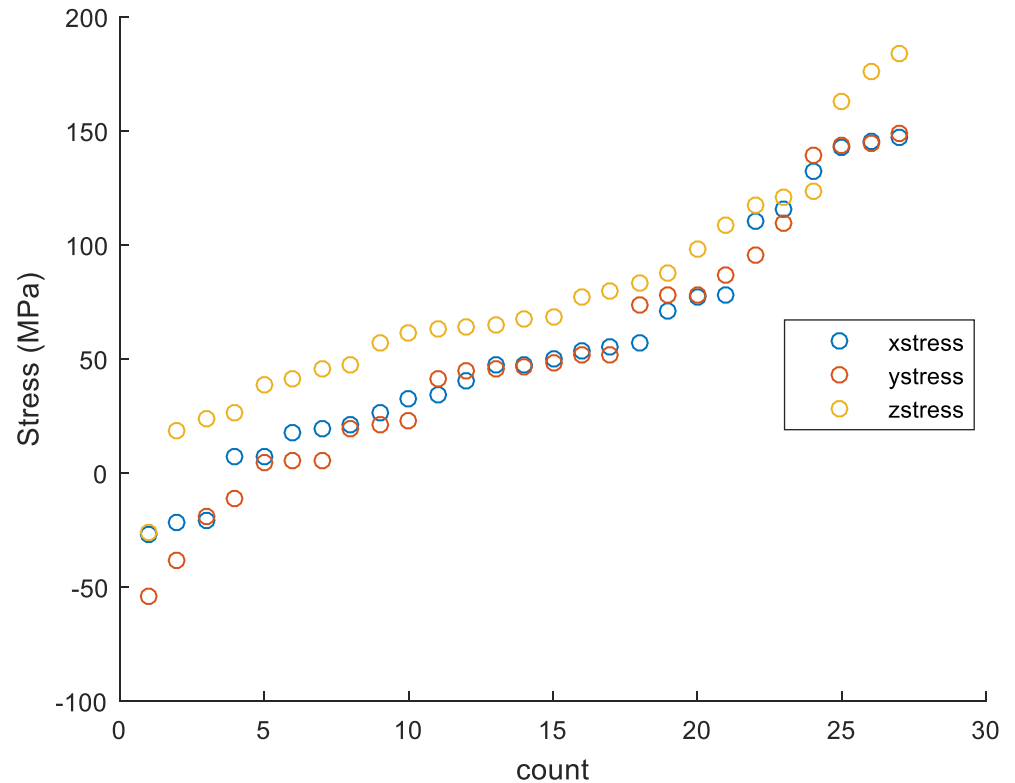


# Volumetric Results

## Example Statistics



4 mm Cube d spacing distribution



10 mm Cube stress distribution

Estimated  $d_0$  – 1.091517 Å

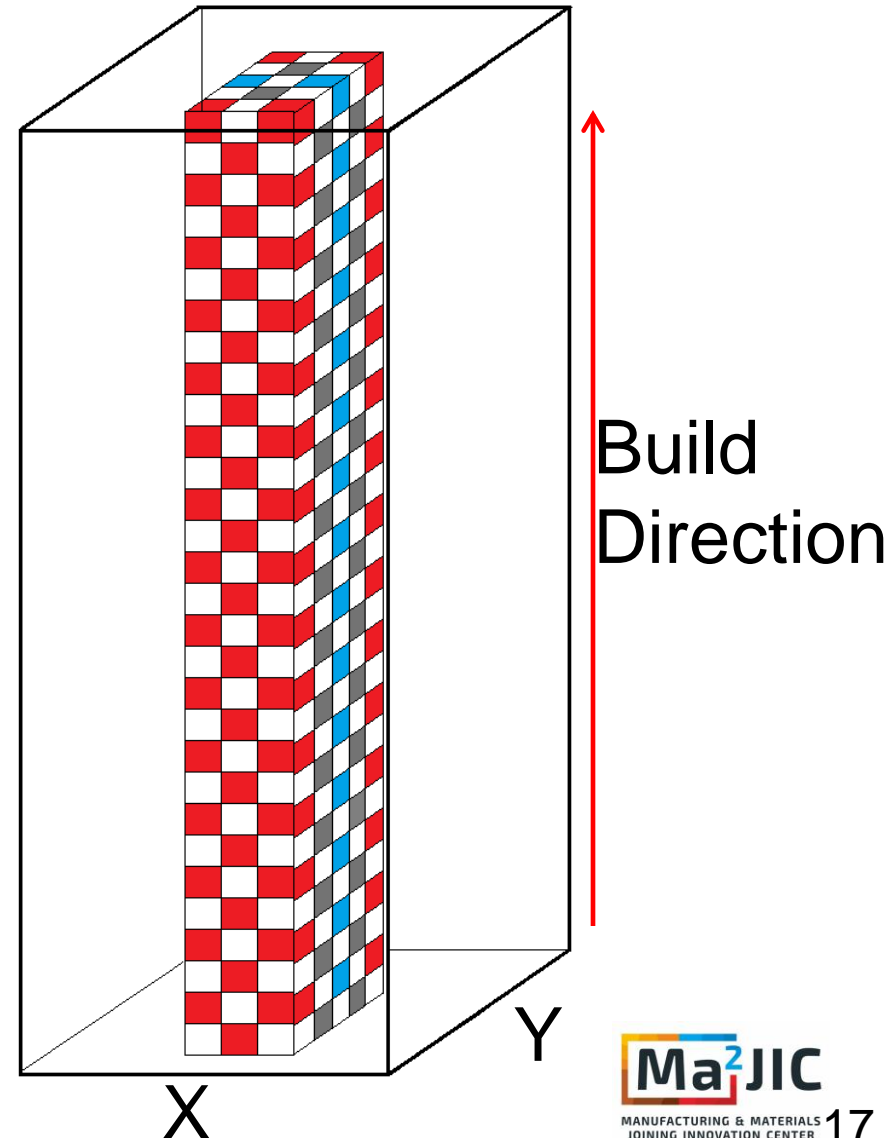


# Pillar Experiment

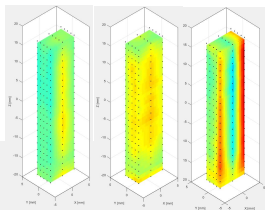
## 2. Pillar Study

- 3x5x22 data points
- Sample goal
  - Function of distance from build-plate

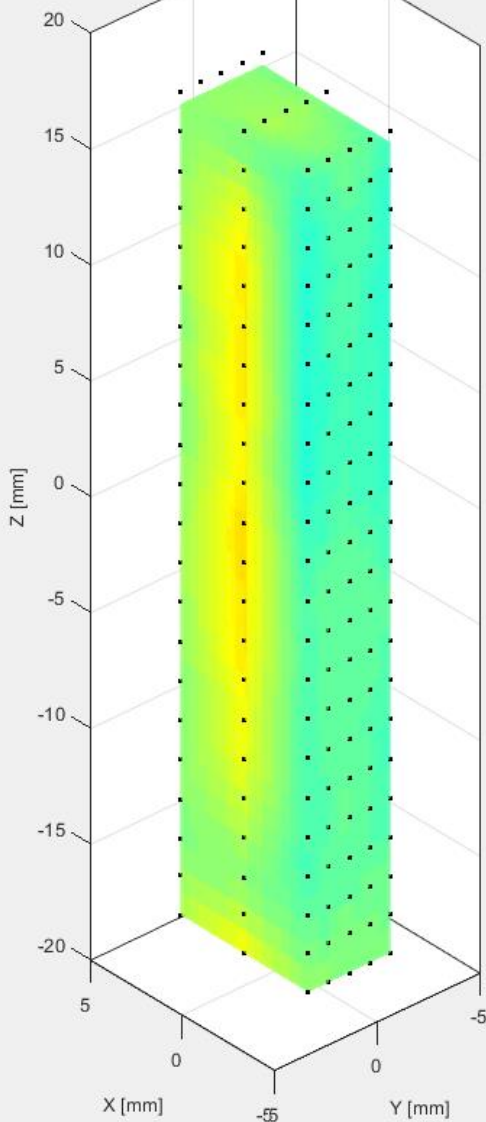
ADD X Y Z Labels



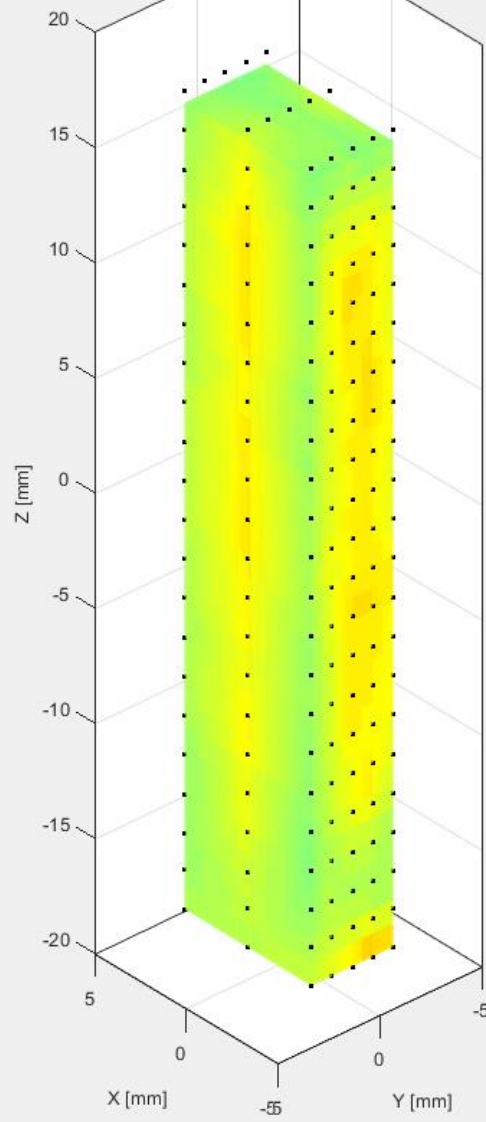
# Pillar Mapping Strain



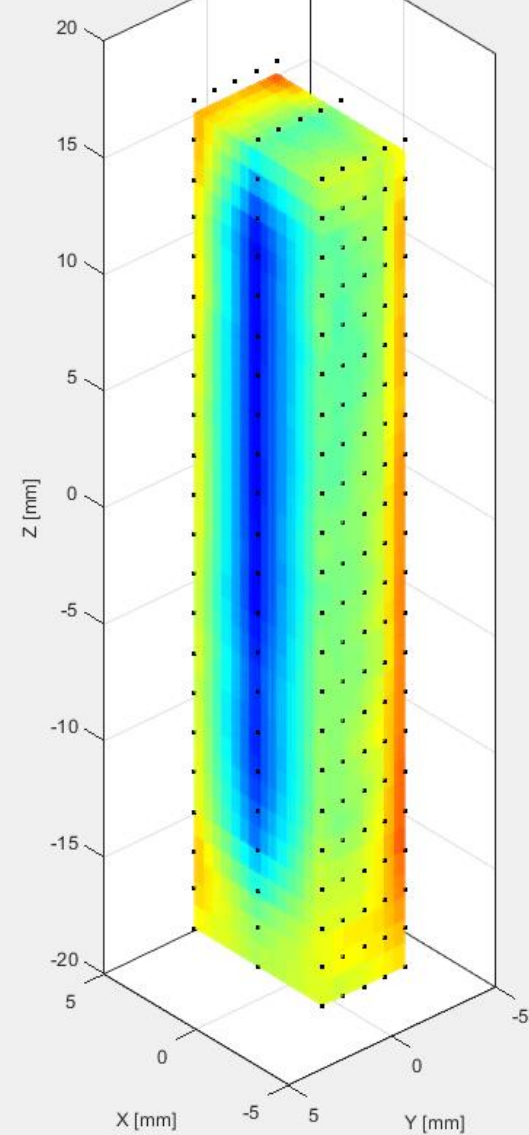
X Direction



Y Direction



Z Direction (Build Direction)



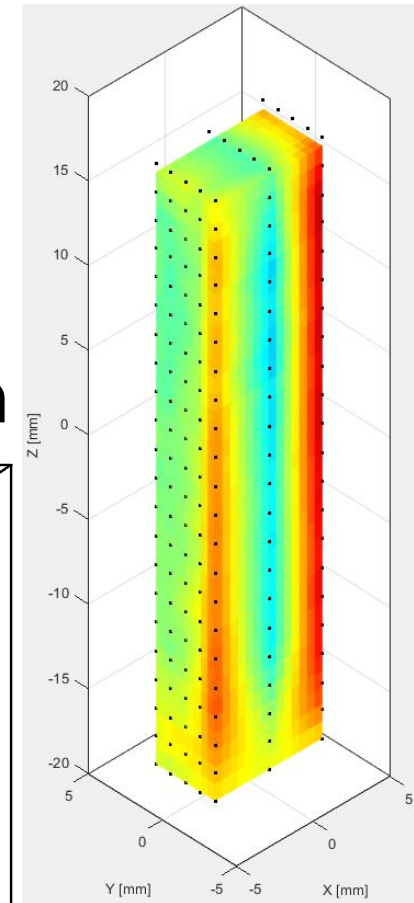
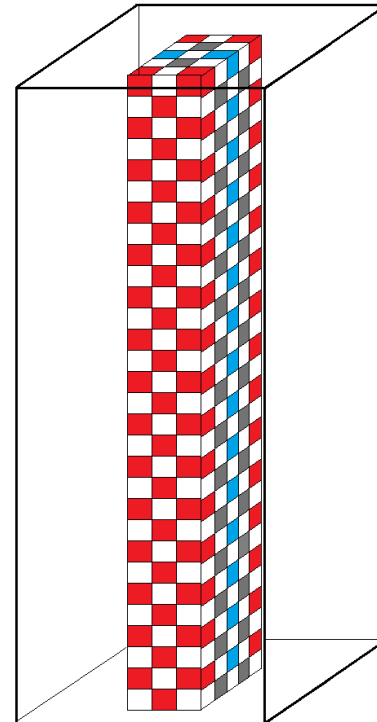
Strain range: 0.00204 to -0.0018





# Pillar Results

- Lit. Elastic Modulus 86 – 111 GPA
  - Being determined by RUSS
    - Resonant UltraSonic Spectroscopy
  - Residual stress could be closer than expected to yield stress
- $d_o$  may change
- Replicate pillar scan
  - with HIPed sample



# Conclusions

## GRCop-84

- Excellent high temp. properties
  - High heat flux applications
- One of the few cases where AM is better than traditional
  - Complex geometry capabilities
  - Lower fabrication time
  - Equal or greater properties
- Relatively easy to print
  - Assumed low RS concentrations

## Neutron Study

- Collected lots of data
  - Stress relieved at interface with build plate
    - Data near bottom needs to account for this
  - 4, 7, and 10 mm cubes
    - Cube samples' better for statistics than mapping
  - 10x10x40 mm pillar
    - HIPed samples to be run
  - +0.00204 to -0.0018 strain
    - Depending on E may be close to yield stress



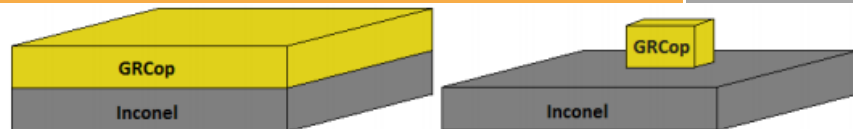
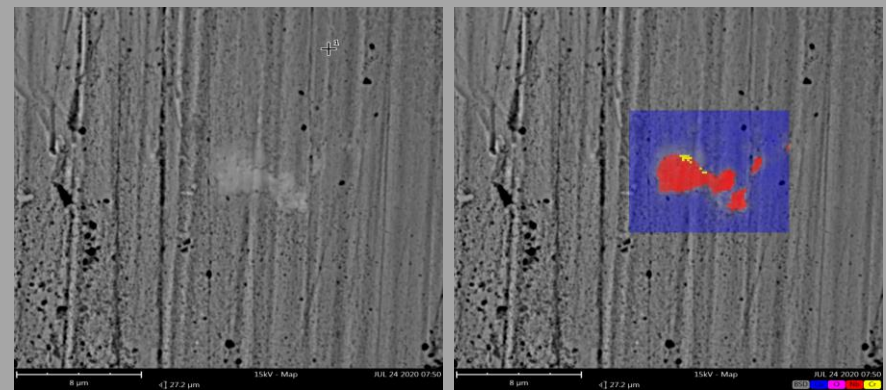
# Future Work

## Next Proposal

- Accepted
  - Sometime later this year
- Thermal and Shape Misfit Analysis
  - GRCo-84 and IN718/625
    - Alloy composite by direct SLM
    - HIPed and unHIPed
  - Finite and semi-infinite cases
  - Difficult  $d_0$  calculation

## Future Objectives

- Complete data analysis
- Characterization
  - SEM
  - EDS
  - Surface XRD
    - HIPed and unHIPed



# References

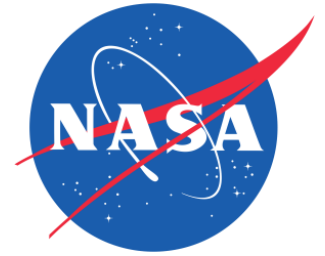
- Ellis, David L. "GRCop-84: A high-temperature copper alloy for high-heat-flux applications." (2005).
- Thoma, D. J., Chu, F., Peralta, P., Kotula, P. G., Chen, K. C., & Mitchell, T. E. (1997). Elastic and mechanical properties of Nb (Cr, V) 2 C15 Laves phases. *Materials Science and Engineering: A*, 239, 251-259.
- Loewenthal, William and Ellis, David. "Fabrication of GRCop-84 Rocket Thrust Chambers." (2005).
- Ellis, D. L., Gray, H. R., & Nathel, M. (2001). Aerospace Structural Materials Handbook Supplement GRCop-84.
- McMahan, Tracy. "NASA 3-D Prints First Full-Scale Copper Rocket Engine Part." *NASA Marshall News*. NASA, 30 July 2015. Web. 13 Dec. 2016. <<https://www.nasa.gov/marshall/news/nasa-3-d-prints-first-full-scale-copper-rocket-engine-part.html>>.
- Izvestiya Akademii Nauk SSSR, Neorganicheskie Materialy (1965) 1, (12) p2205-p2207 Cr2Nb ICSD 102795 Data
- Journal of Applied Physics (1961) 32, p1536-p1546 Cu ICSD 43493 Data
- Carter, Robert, et al. "Evaluation of GRCop-84 Produced Using Selective Laser Melting." White paper (2015).
- Carter, Robert, et al. "Materials Characterization of Additively Manufactured Components for Rocket Propulsion." (2015).
- "Neutron Scattering Lengths and Cross Sections." Neutron Scattering Lengths and Cross Sections. NIST, 07 Jan. 2013. Web. 27 Sept. 2016.
- Bagg, Stacey D., Lindsay M. Sochalski-Kolbus, and Jeffrey R. Bunn. "The Effect of Laser Scan Strategy on Distortion and Residual Stresses of Arches Made With Selective Laser Melting." (2016).
- Eisazadeh, Hamid, et al. "A residual stress study in similar and dissimilar welds." *Welding Journal* 95.4 (2016).





THE UNIVERSITY OF  
TENNESSEE  
KNOXVILLE

COLLEGE OF ENGINEERING



# Thank you

# Questions?



RPM would like to acknowledge partial support through the Manufacturing and Materials Joining Innovation Center (Ma2JIC) University of Tennessee, Knoxville site. Ma2JIC is funded by the National Science Foundation (NSF) through the Industry/University Cooperative Research Center (I/UCRC) program award number IIP 1540000.

This research [or, A portion of this research] used resources at the High Flux Isotope Reactor [and/or Spallation Neutron Source, as appropriate], a DOE Office of Science User Facility operated by the Oak Ridge National Laboratory.

