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Experimental Investigations of an Additively Manufactured Multi-Principal Element Alloy with Extraordinarily High Strength

Morgan Jones[1,2], Eric Hintsala[4], Douglas Stauffer[4], Jonathan Pegues[2], Ping Lu[2], Frank DelRio[2], Iver Anderson[3], Duane Johnson[3], Andrew Kustas[2], Nicolas Argibay[3], and Irene Beyerlein[1]

[1] Materials Department
University of California, Santa Barbara
Santa Barbara, CA

[2] Materials Science and Engineering Center
Sandia National Laboratories
Albuquerque, NM

[3] Materials Science Center
Ames Laboratory
Ames, IA

[4] Bruker Nano, Inc
Eden Prairie, MN

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A New Frontier for Alloy Discovery and Design

Superalloy for Structural Applications Wishlist:

high strength & toughness

good ductility

corrosion & erosion resistant

wear & oxidation resistant

high creep & fatigue strength

easy to process

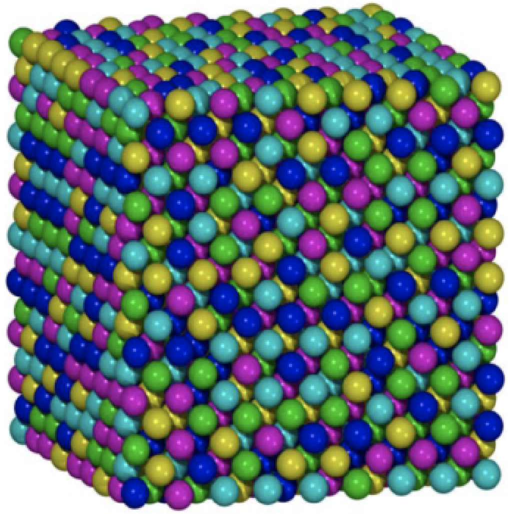
retention of strength at high temperatures

FCC CoCrFeMnNi (Cantor) Alloy

Exceptional damage tolerance (K_{JIc}=200 MPa-m$^{1/2}$), high strength (σ_y =362 MPa), and ductility (ε =51%)

BCC HfNbTaTiZr (Senkov) Alloy

Good processability, high strength (σ_y = 929 MPa) and ductility (ε =50%), impressive σ_y up to T ~ 1000 ºC

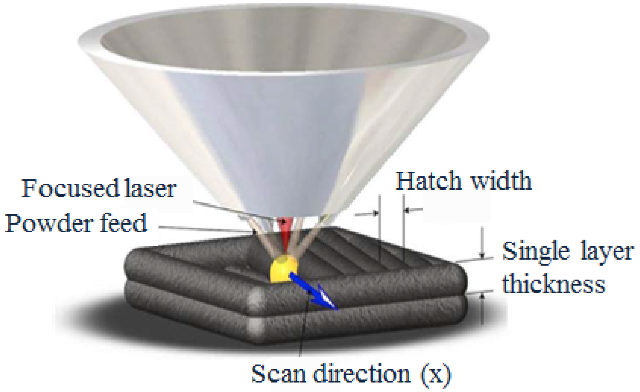


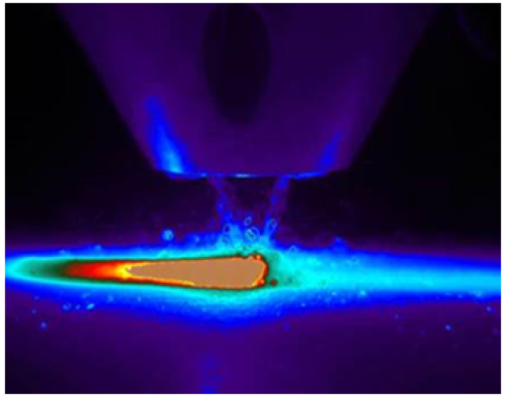
Atomic structure model of CoCrFeMnNi

(George, et al., Nature Rev., 2019)
(Miracle and Senkov, Acta Mat, 2017

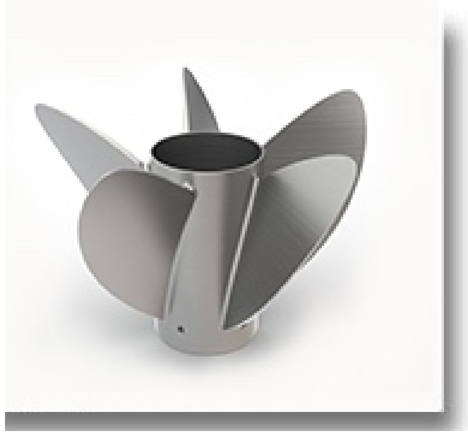
Realizing the Potential of AM

Long-Term Goal: Enable tools like SNL's LENS (Laser Eng. Net Shaping)…

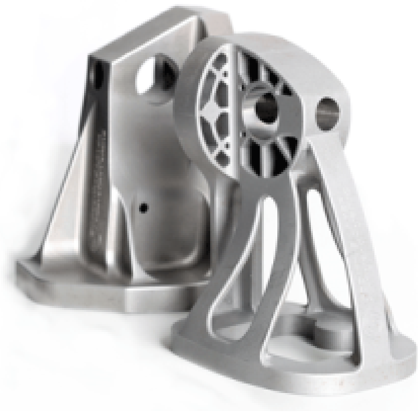




… to make "born qualified" parts (i.e., reliably high strength, ductility, etc):



Source: https://www.sciaky.com/additive-manufacturing/contract-additive-manufacturing-services

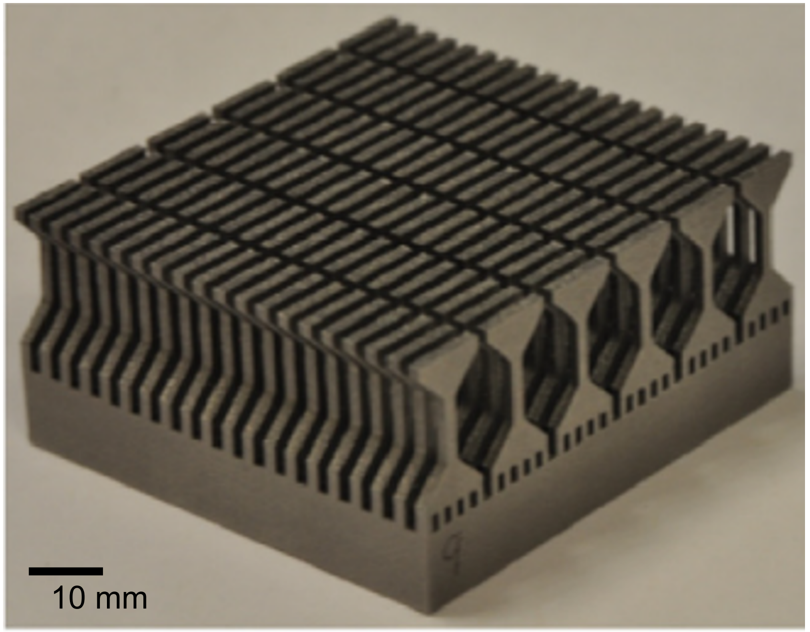


Prototype Airbus A380 bracket

Source: https://www.metal-am.com/introduction-to-metal-additive-manufacturing-and-3d-printing/

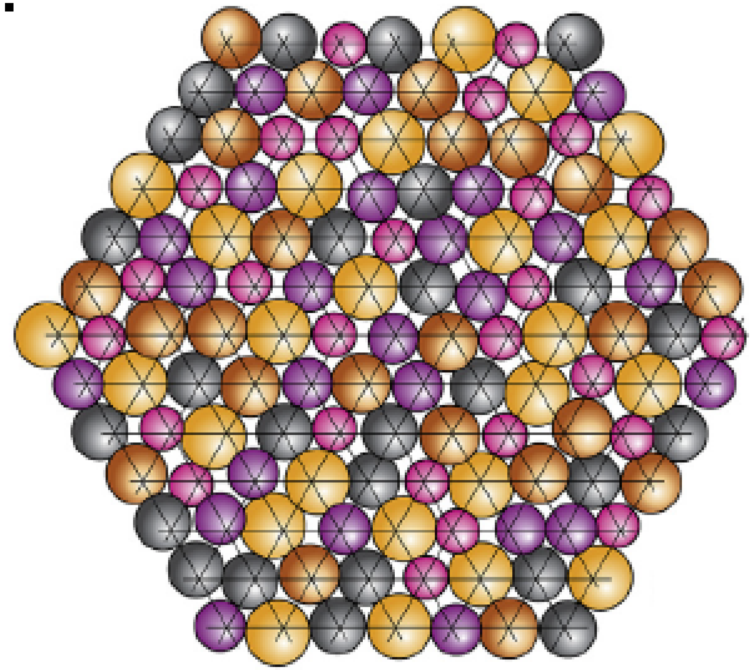
Why are we Printing MPEAs?

Conventional alloys degrade from melt/re-melt solidification processes.



Laser consolidated 17-4 PH Steel: Reduced strength AND ductility attributed to higher surface roughness, porosity, and microstructure.

Conversely… MPEAs can derive much of their strength from solid solution strengthening, and are **less sensitive to processing conditions (i.e., thermal history).**



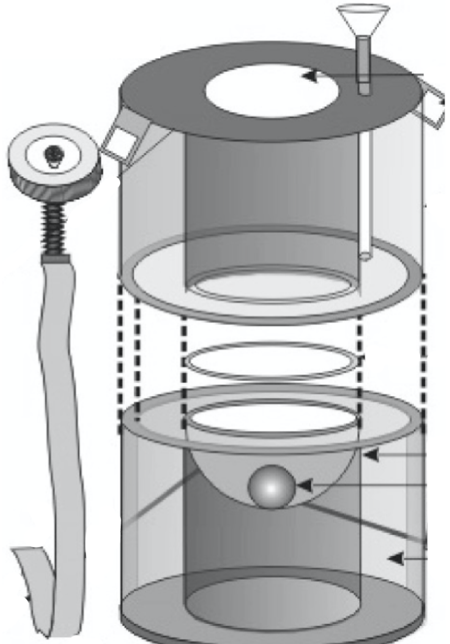
(Miracle, et al., Acta Mat., 2017

(B. Salzbrenner, et al., J. Mat. Proc. Tech., 2017)

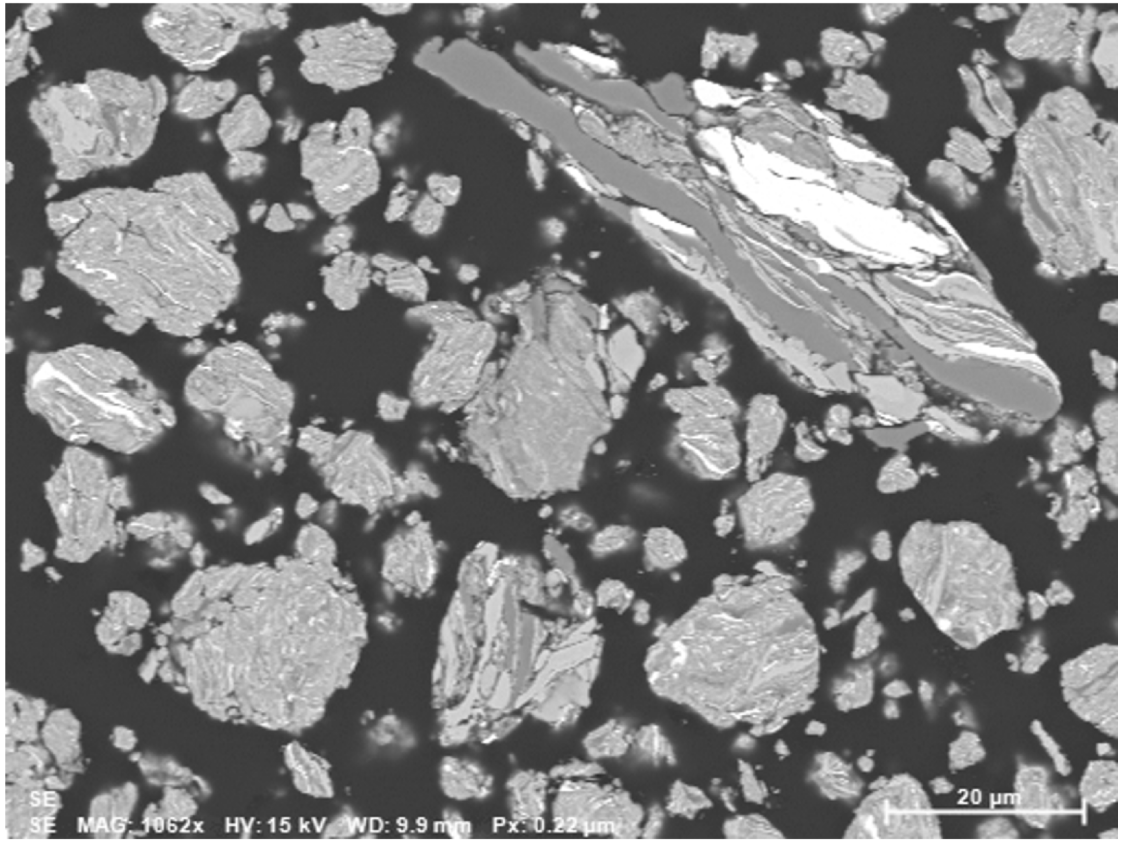
$Al_{0.42}Mo_{0.08}Nb_{0.13}Ta_{0.04}Ti_{0.25}Zr_{0.08}$ MPEA ("Kustalloy")

powder was initially elementally segregated

mechanically mixed (cryo-ball milling)

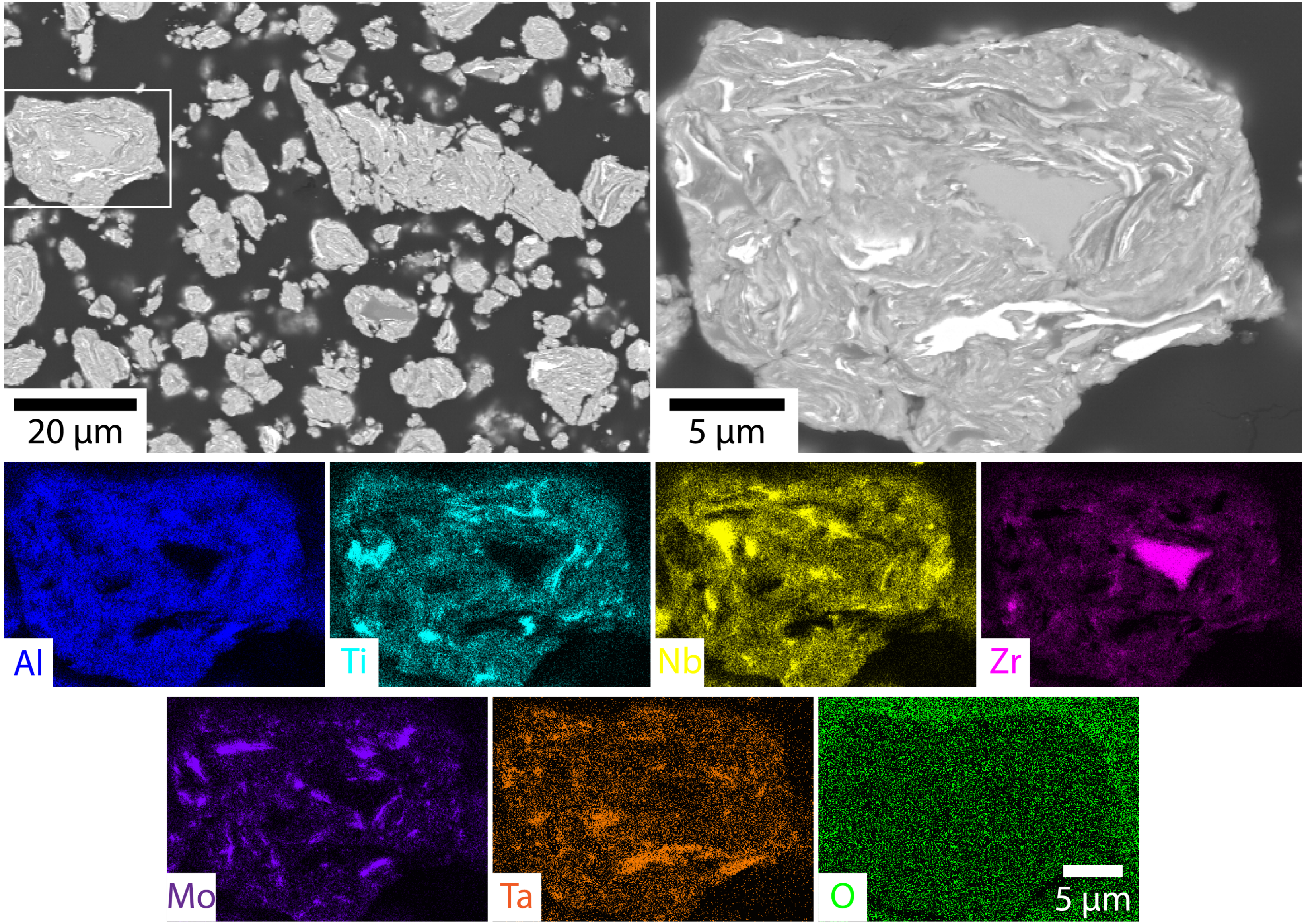


...and got this: suboptimal morphology, lack of spheroidicity, satellites, poor flowability



(Kumar, et al., Rev. Sci. Inst., 2015)

$Al_{0.42}Mo_{0.08}Nb_{0.13}Ta_{0.04}Ti_{0.25}Zr_{0.08}$ MPEA ("Kustalloy")



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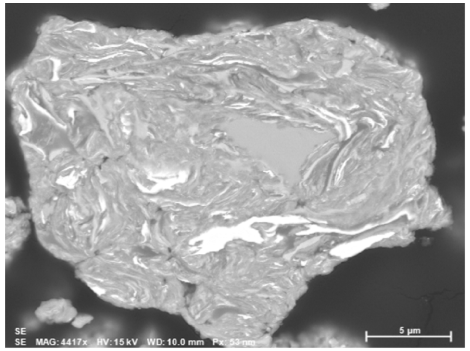
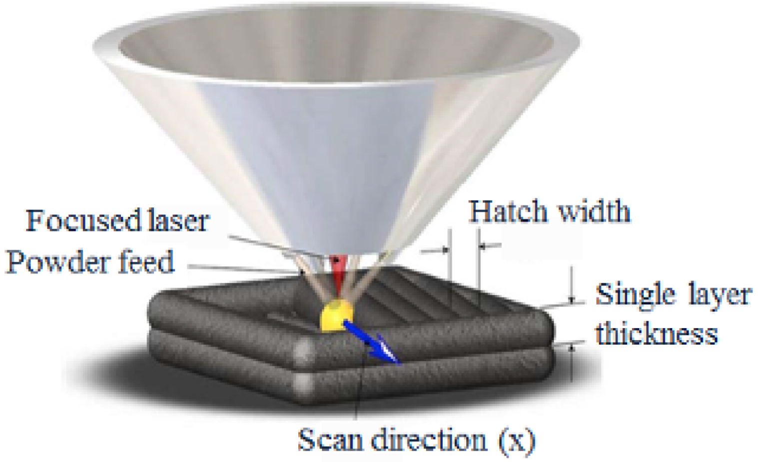


how? sintering kicks off highly exothermic reaction; calculated values of formation energy are massive and negative

despite morphology, took very little laser power to begin alloying

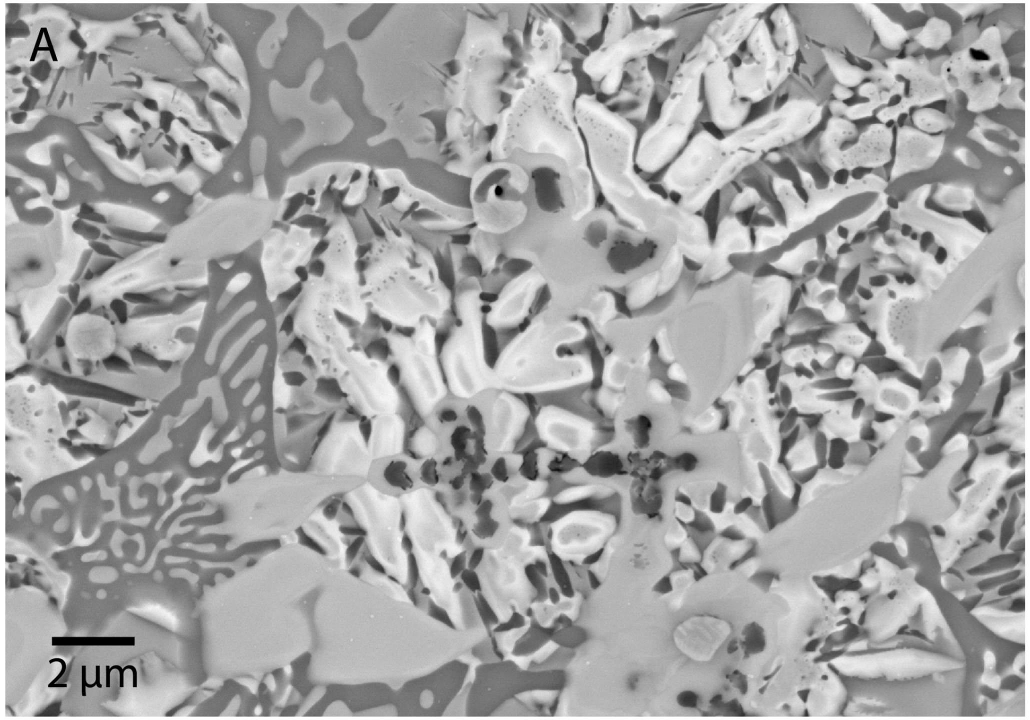


powder consolidated with only 100 W!

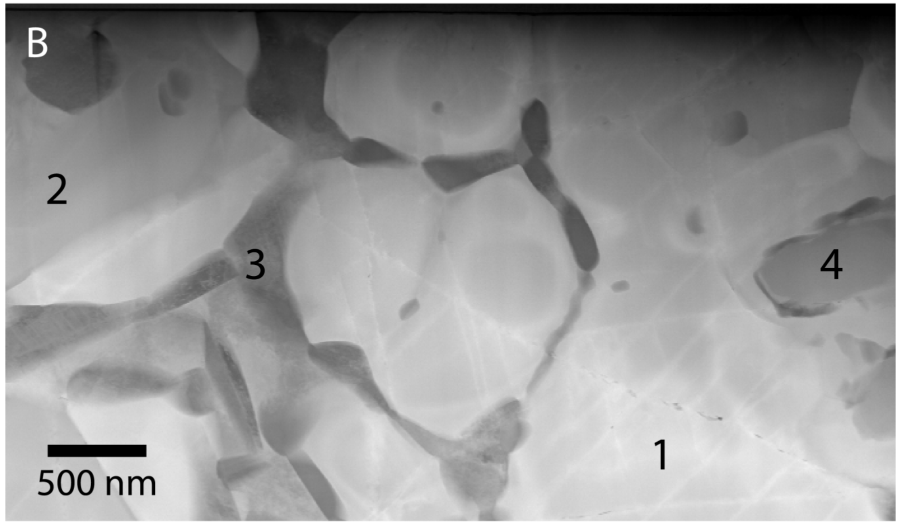
Even with the refractory metals, microscopy does not show signs of porosity or partial melting!

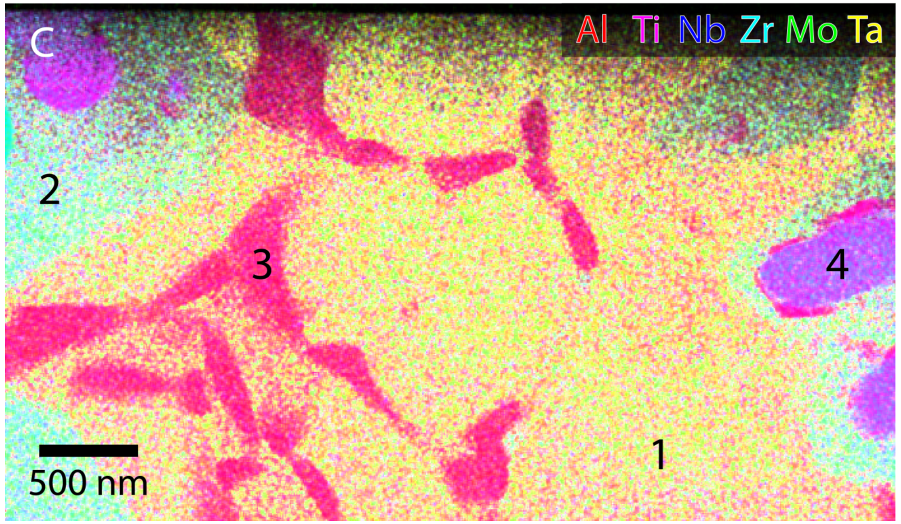
Multi-phase, Multi-modal Grain Size Distribution

SEM:



STEM with Chemical Mapping:

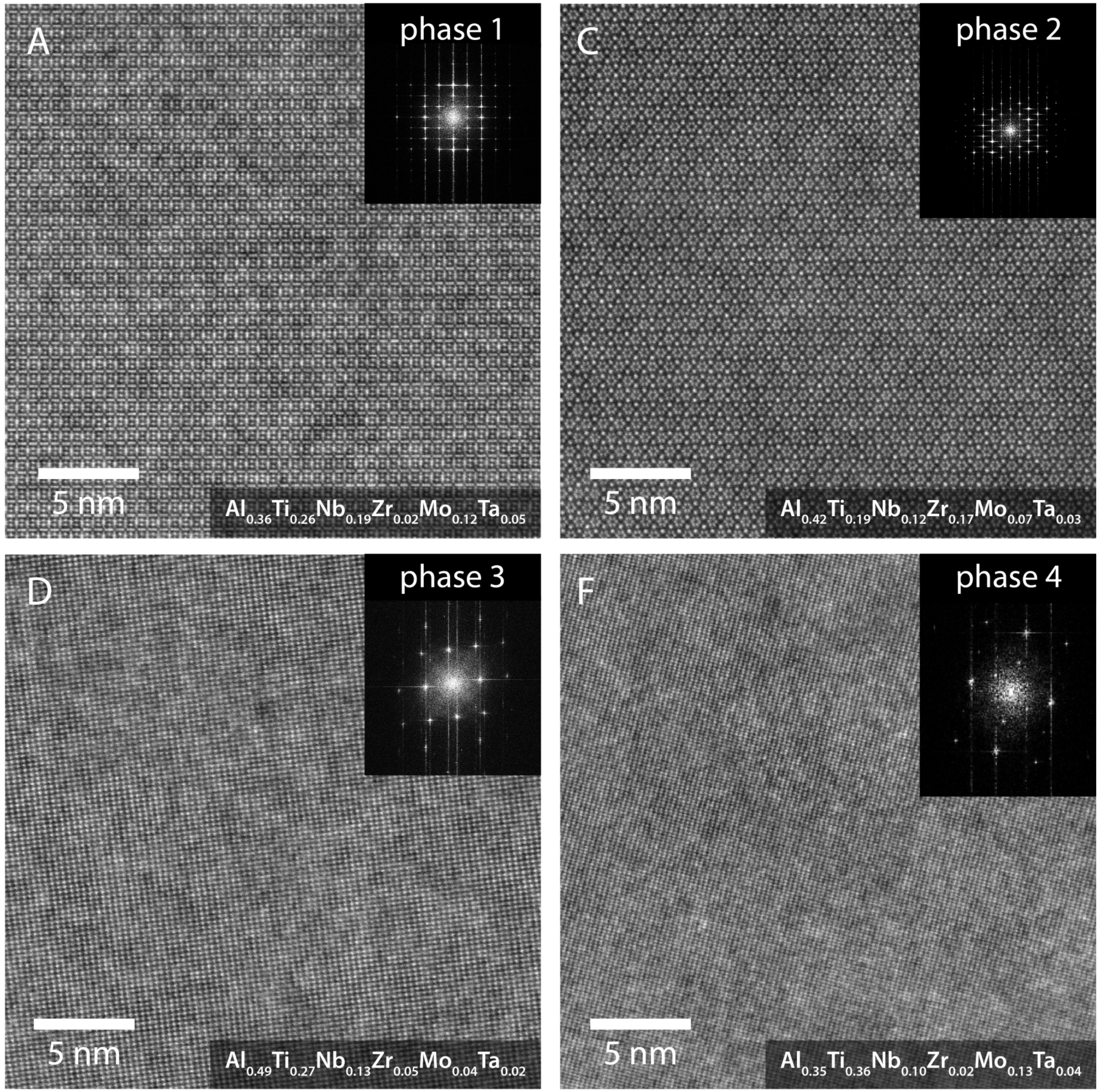




SEM shows grain sizes range from 10s of nm to 10s of μm

STEM shows presence of four distinct phases

Four Distinct Phases

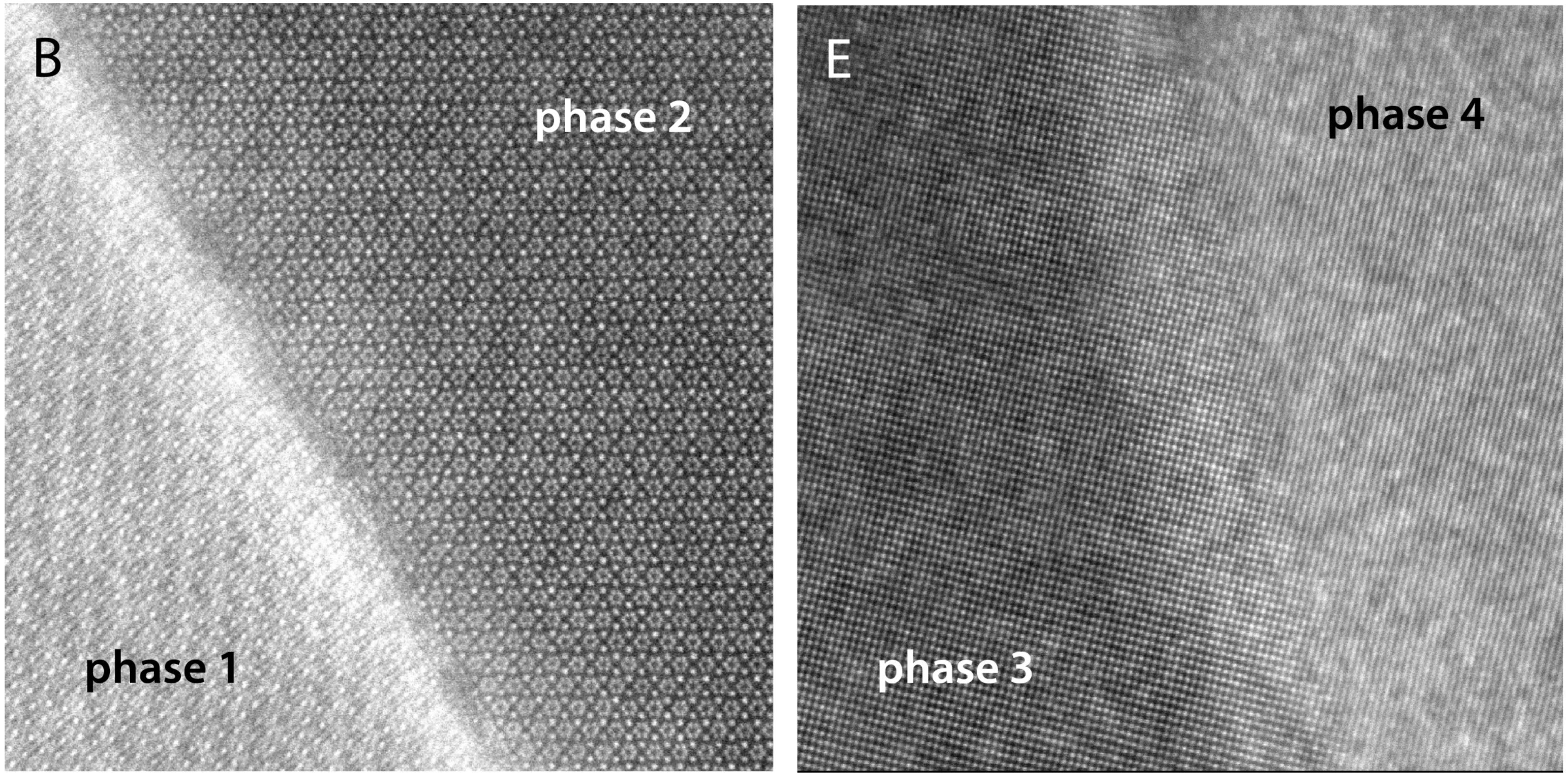


STEM-HAADF images and FFT patterns of the images for the four distinct phases.

Despite extensive investigation (XRD, Precession electron diffraction, Synchrotron, STEM chemical mapping)…

Previously unidentified crystal structures were present in the material, suggesting follow-on opportunities for identification of these new structures.

With Highly Coherent Phase Boundaries



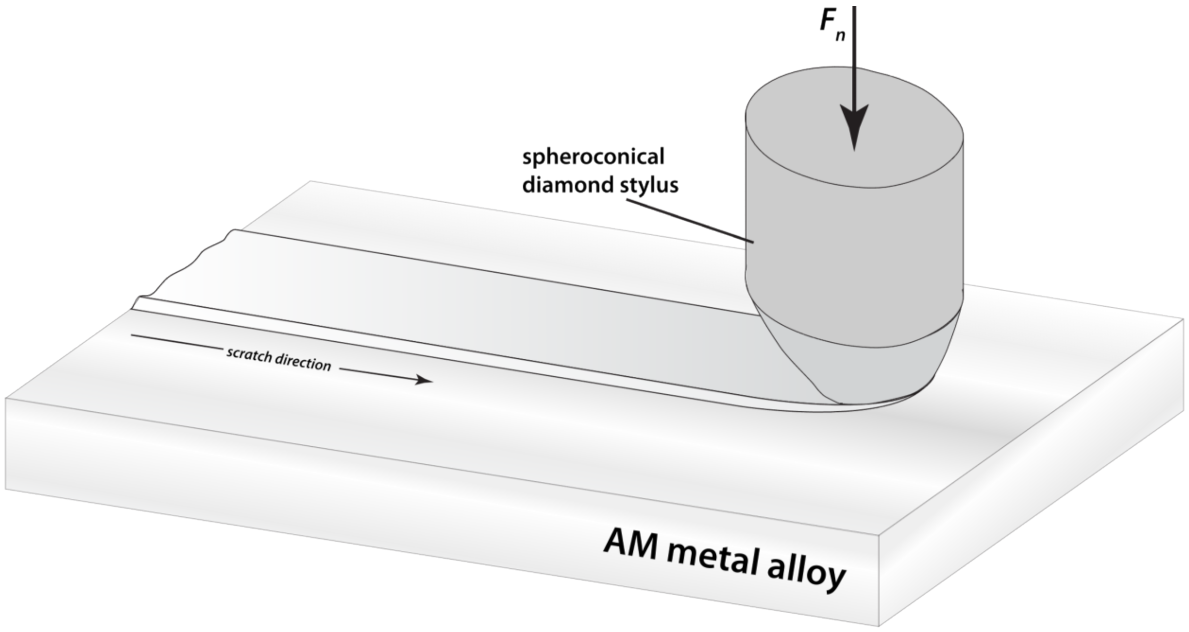
Evidence that some interphase boundaries are highly coherent, with misorientation of less than 2° between phases 1/2 and 3/4.

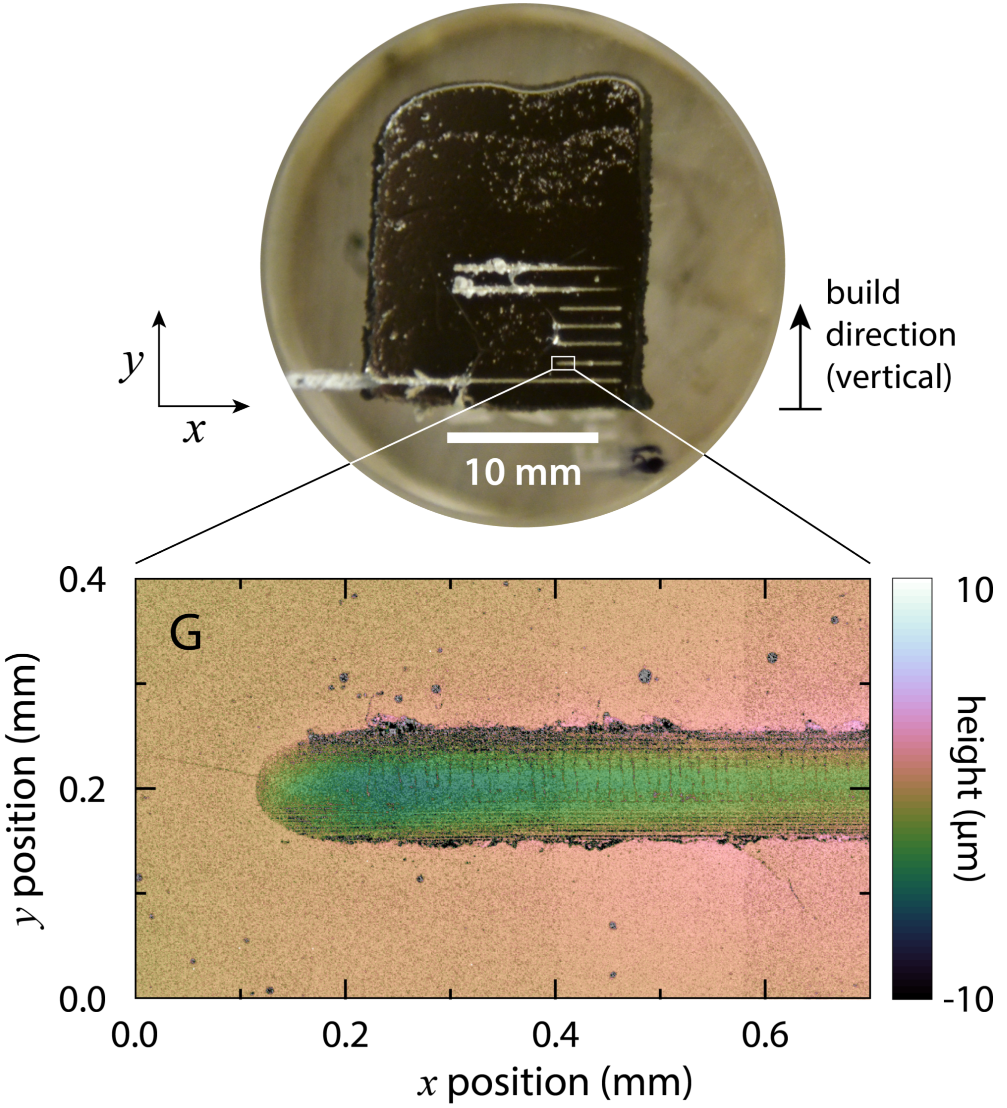
Literature on coherent phase boundaries:

Exhibit atypical combination of high strength and ductility

Promotes dislocation accumulation while mitigating crack formation/propagation

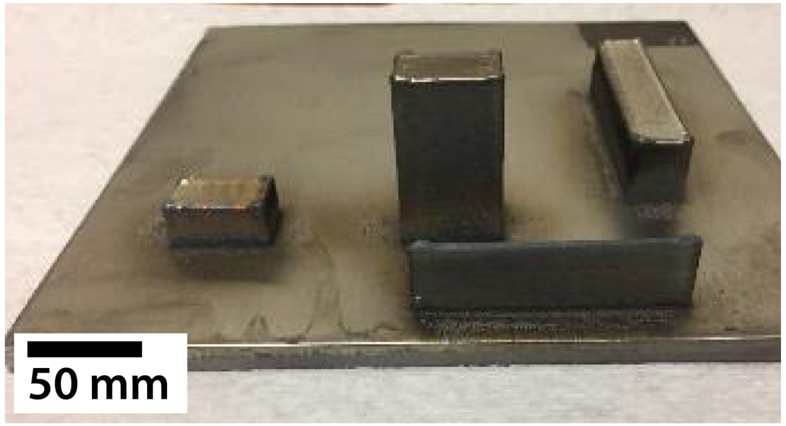
Utilizing Surface-Based Deformation Techniques

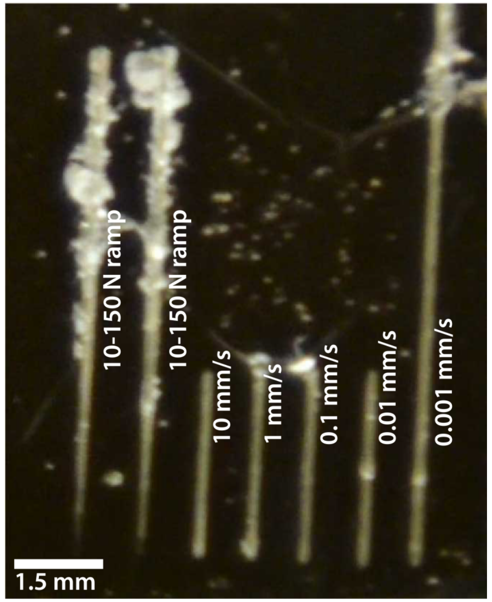


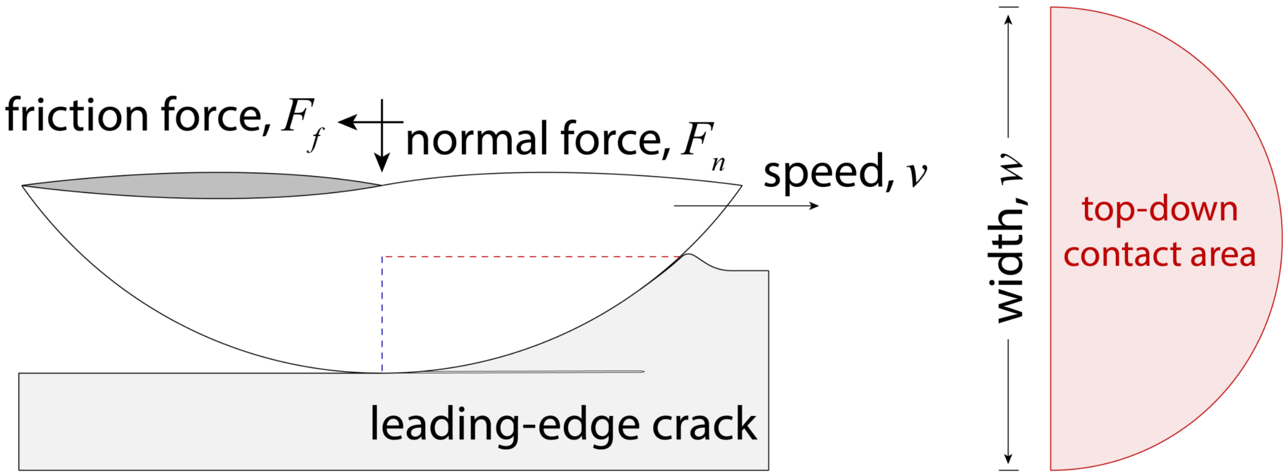


High-throughput characterization possible even on ‘bad’ AM specimens; voids, dimples, cracks would make this specimen unusable for tensile testing, but all we need is a polished surface.

Scratch Testing for Strain-Rate Dependent Hardness

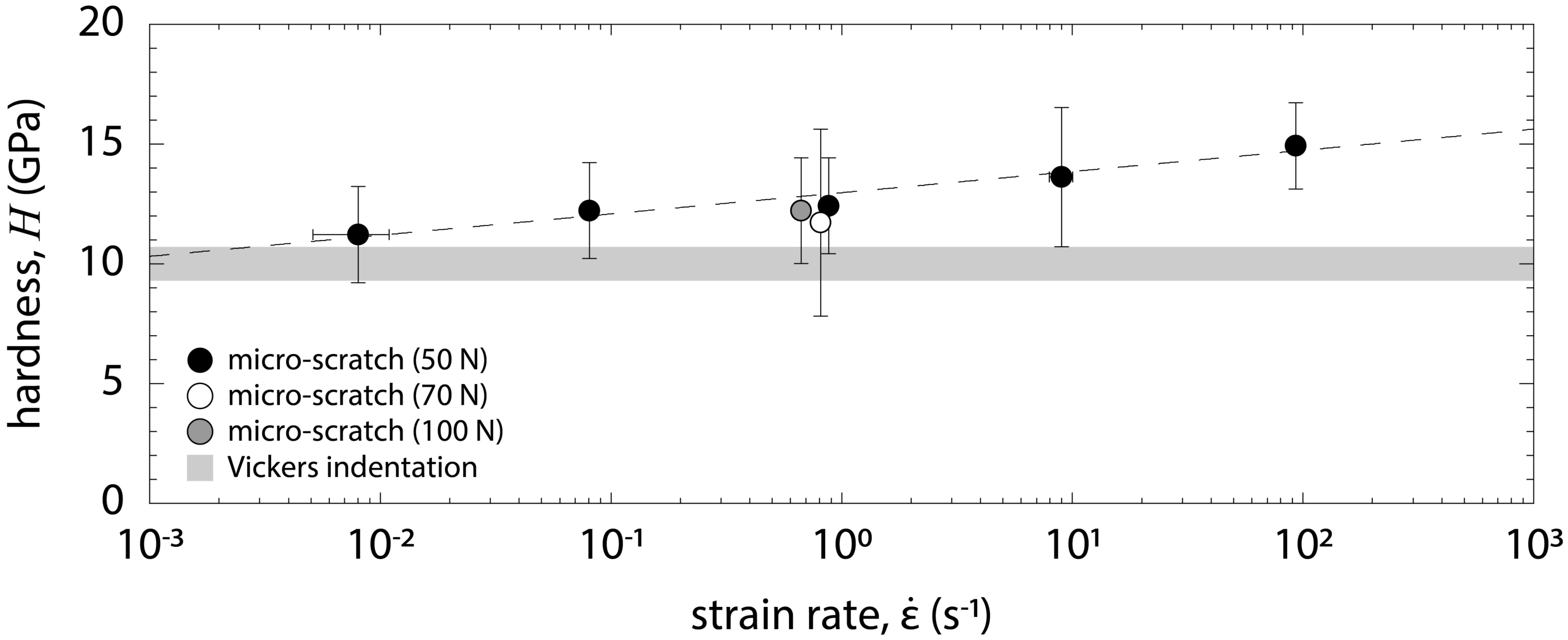






strain rate (s^{-1})	hardness (GPa)

Kustalloy Strain-Rate Dependent Hardness



Kustalloy exhibits > 2x the hardness value of Inconel 718, which has a reported peak hardness of ~4.5 GPa.

SRS and Activation Volume

strain-rate sensitivity exponent (SRS)

activation volume

$m \cong 0.03$

$V^* \cong 55\ A^3 \cong 1.67b^3$

SRS value is consistent with fine-grained (d $\cong$ 1 μm) bcc & fcc pure metals

In the context of MPEAs, SRS value falls in about the middle of reported ranges ($m \cong 0.01 - 0.06$)

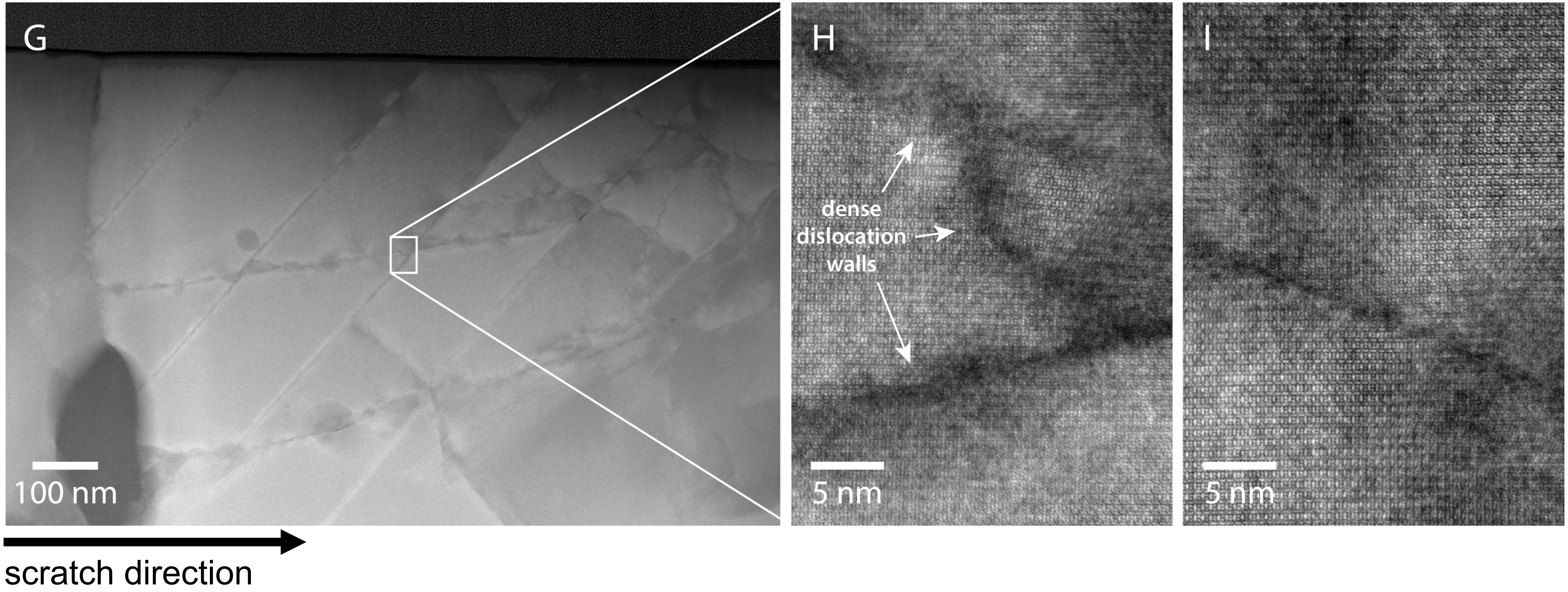
V* value that ranges from $1b^3 - 10\ b^3$ usually indicates that a Peierls-Nabarro mechanism is dominant

(Wei, et al., Matl Sci & Eng A, 2004)

(Lilensten, et al., Acta Mat, 2018)

(Xiao, et al., Matl Sci & Eng A, 2020)

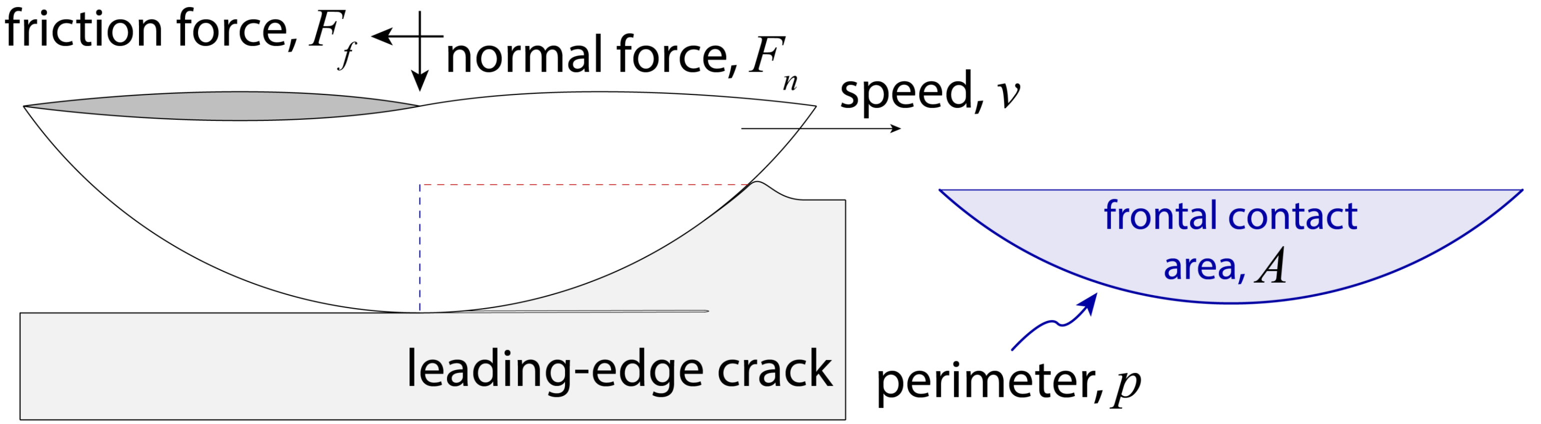
TEM Images of Scratch Track



Negligible change in grain size inside scratch track

Images suggest plasticity was accommodated by intragranular deformation and the formation of dislocation walls

Also Possible to Determine Mode II Fracture



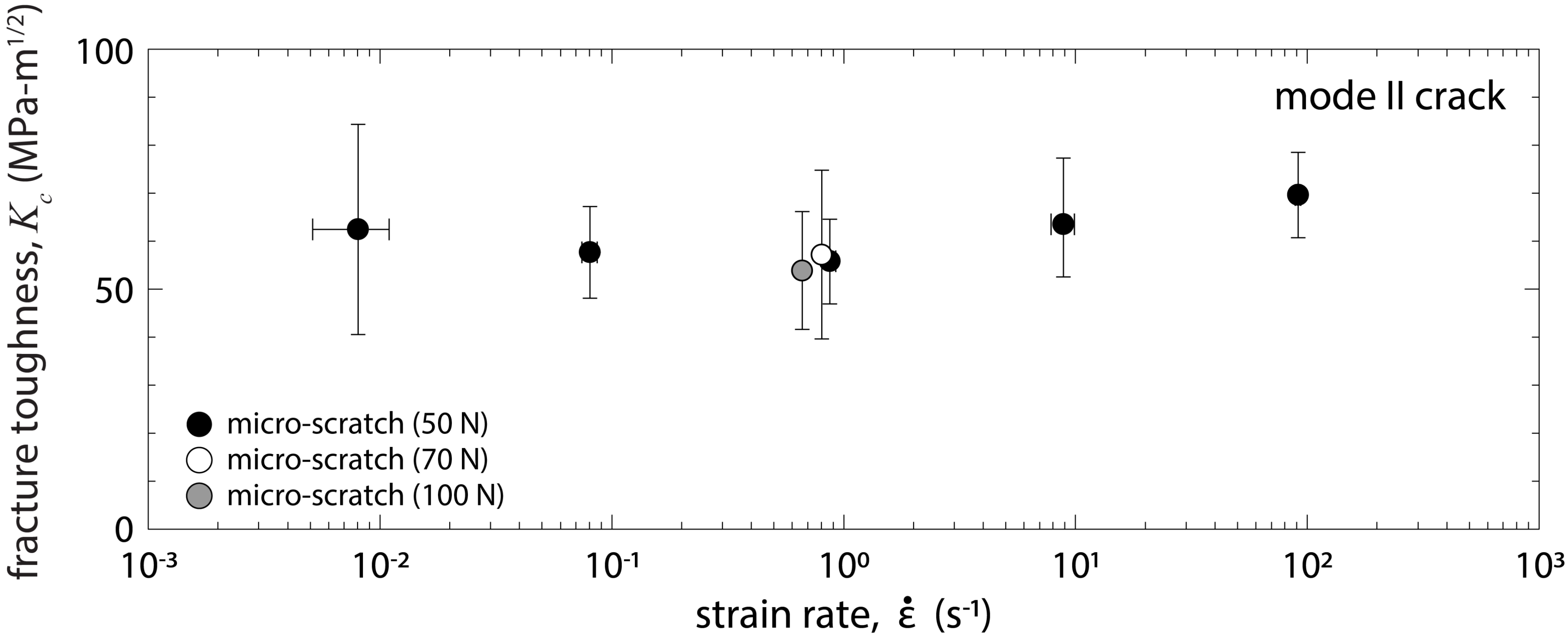
$$K_c = \frac{F_t}{(2pA)^{1/2}}$$

Technique extracts fracture toughness from measured forces and penetration depth during a scratch test with a Rockwell (sphero-conical) probe.

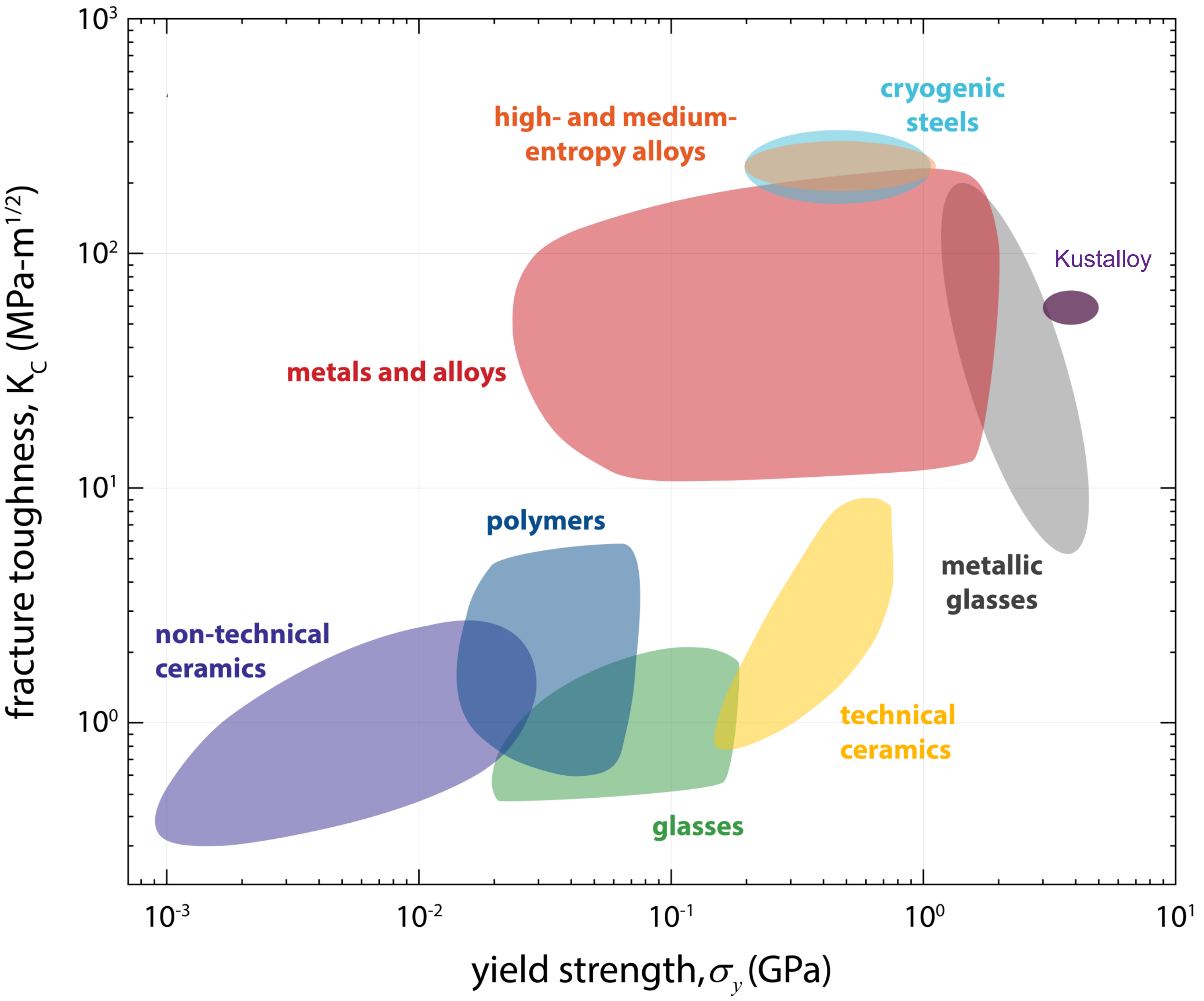
Experimental determination of the fracture toughness via microscratch tests: Application to polymers, ceramics, and metals

(Akono, et al., J Matl Res, 2012)

Kustalloy Strain-Rate Dependent Fracture Toughness



Ashby Plot



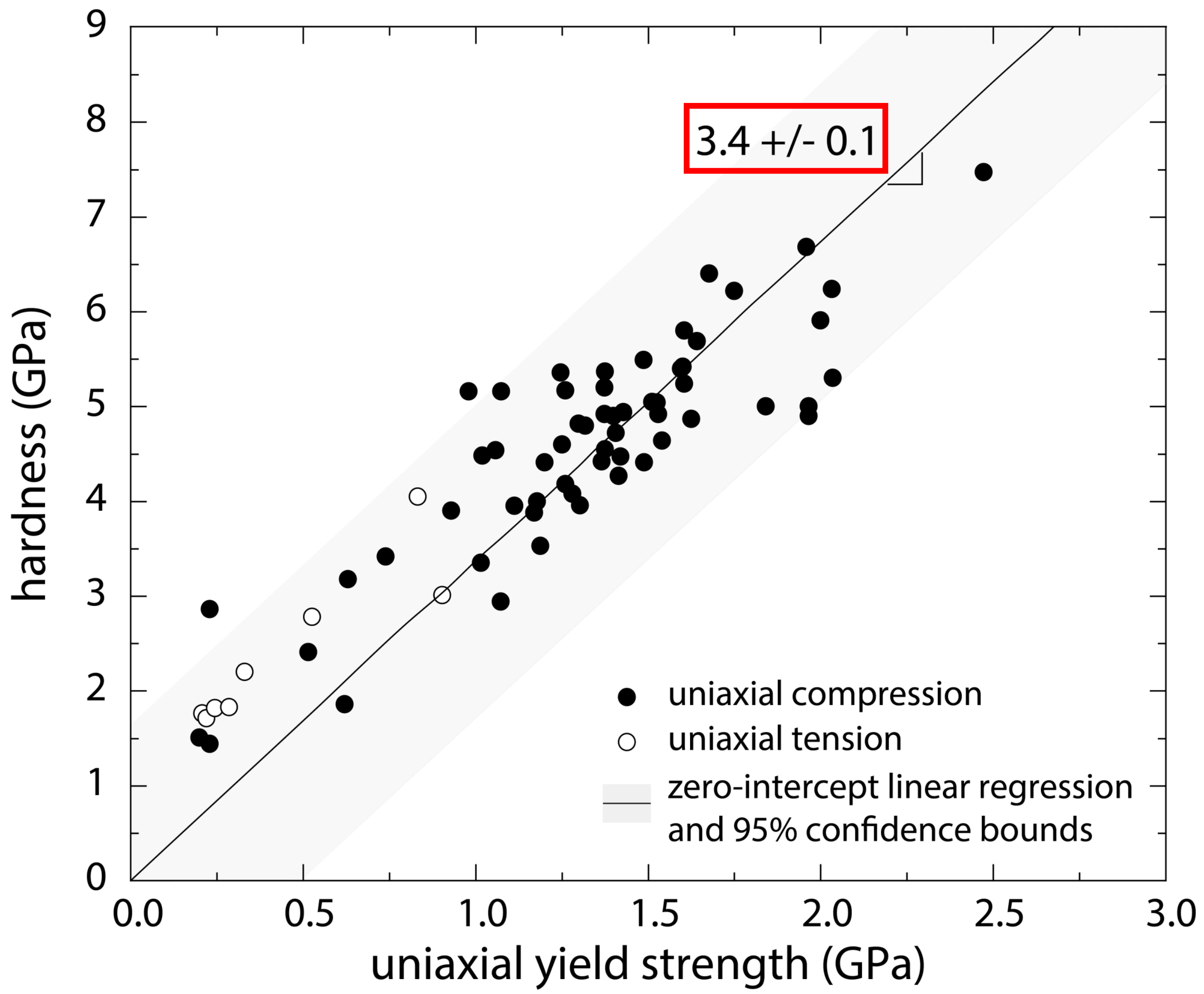
Tabor Relation:

$$H \cong 3\sigma_y$$

Ashby plot of strength vs. fracture toughness shows our data (purple) are among the most damage-tolerant and high-strength on record.

(adapted from: George, Nature Mat Rev, 20

Tabor Relation for MPEAs?

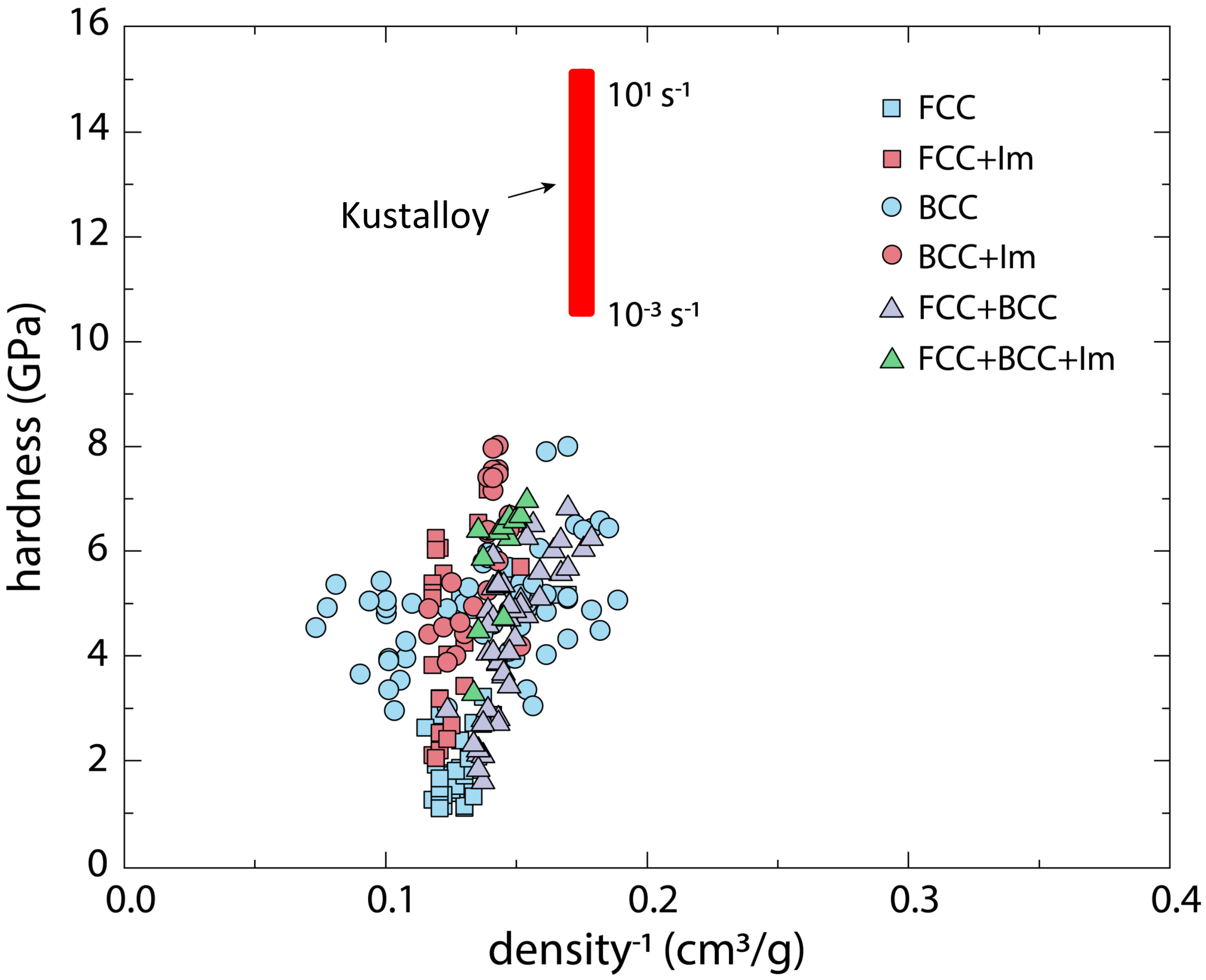


From database of mechanical properties, plotted reported hardness vs. reported strength for MPEAs that had both values listed.

Slope is slightly higher than what would be predicted by Tabor relation, but not far off!

literature from (Gorsse et al., Data in Brief, 20

Comparison with State-of-the-Art



Kustalloy density:

Archimedes method, $\rho = 5.7\ g/cm^3$

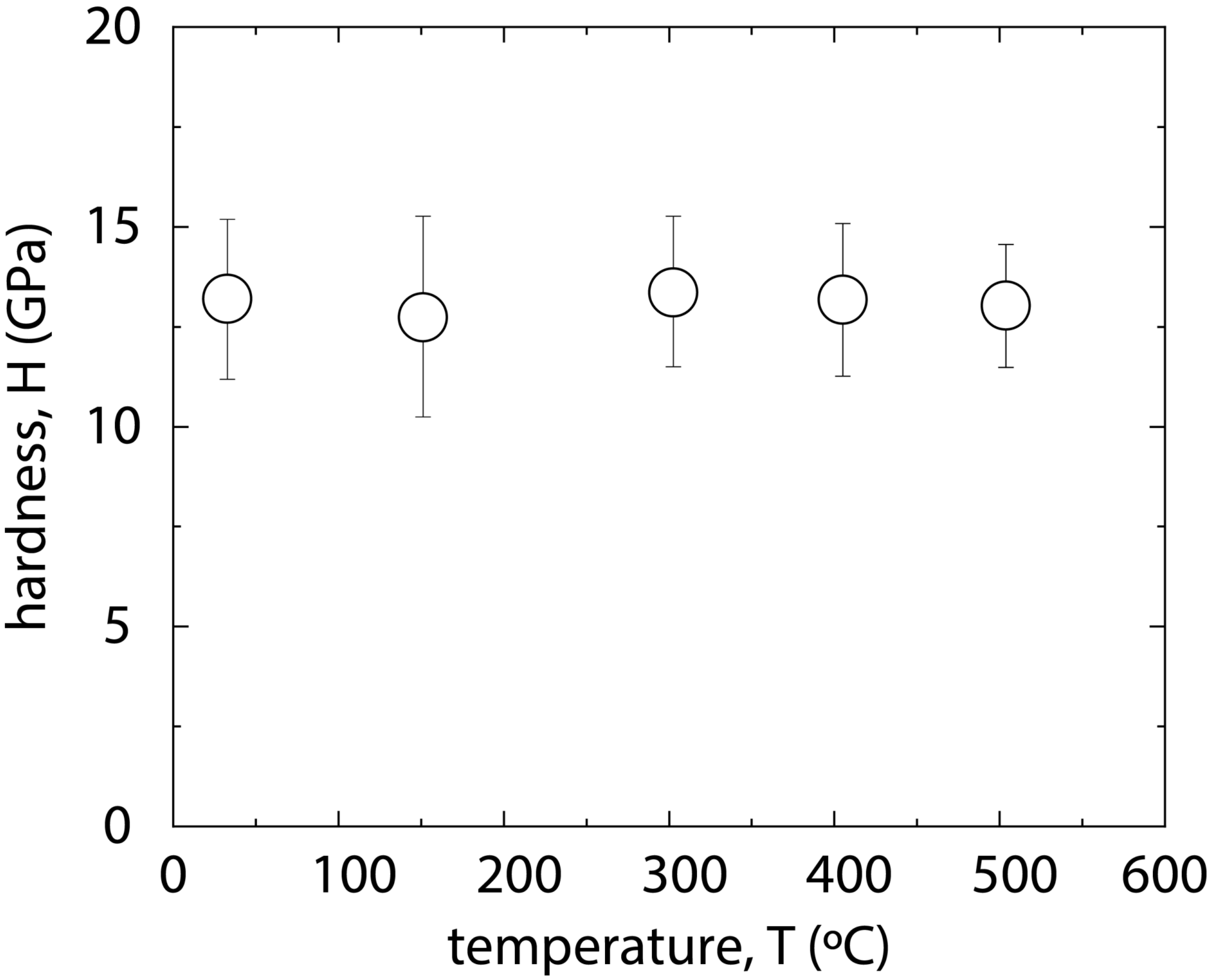
DFT calculation, $\rho = 5.4\ g/cm^3$

Plotted H vs. reported $\boldsymbol{\rho}$ for MPEAs that had both values listed.

notice x-axis plotted 1/$\boldsymbol{\rho}$

literature from (Gorsse et al., Data in Brief, 20

Kustalloy High-Temperature Hardness

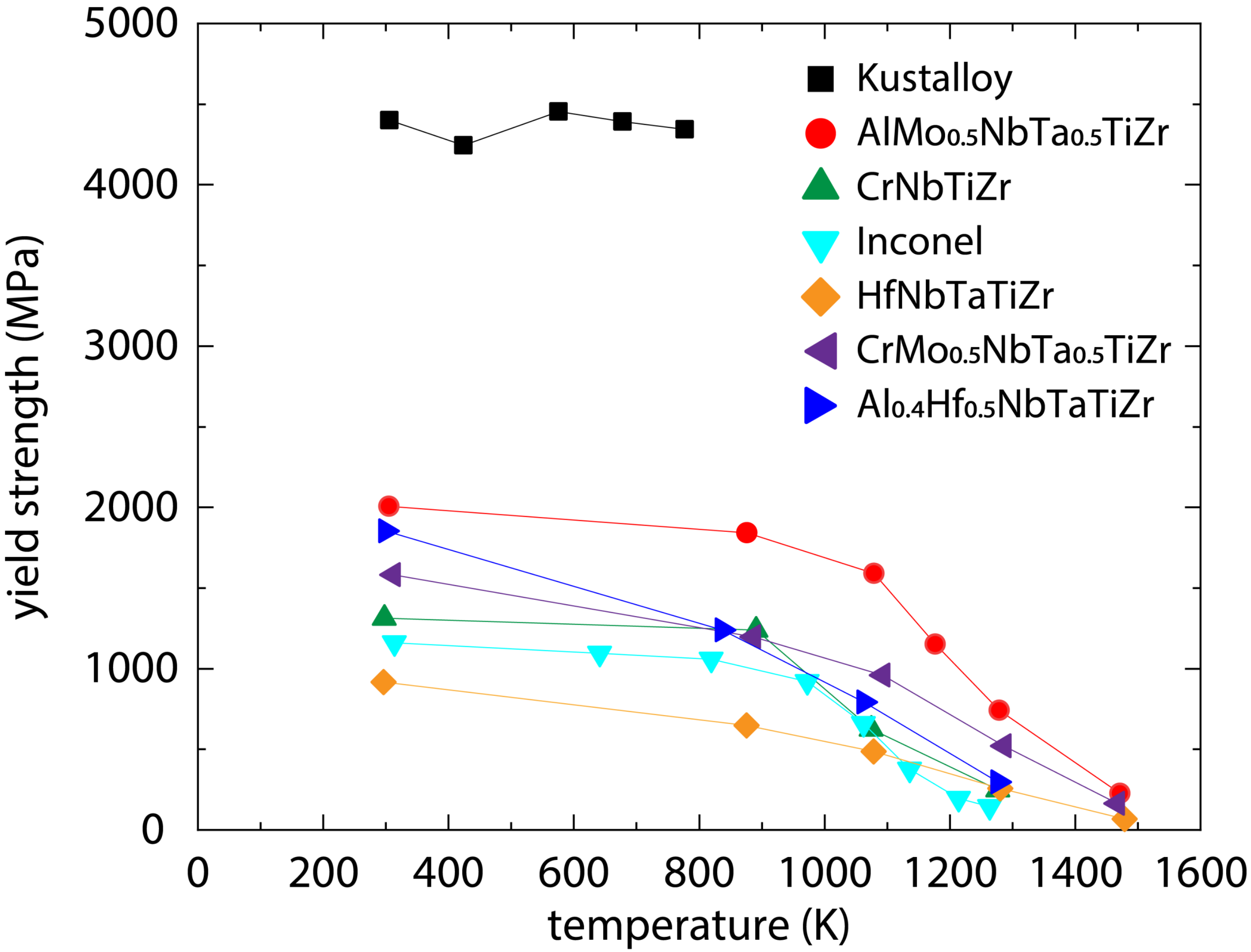


High-temperature nanoindentation technique: 10 x 10 indent array at each temperature point using XPM

Retention of strength up to 500 ^{o}C

Repeating the experiment to populate 600 ^{o}C, 700 ^{o}C, and 800 ^{o}C data points… stay tuned

Comparison with State-of-the-Art



Tabor Relation:

$$H \cong 3\sigma_y$$

Recall constituents of Kustalloy:

$Al_{0.42}Mo_{0.08}Nb_{0.13}Ta_{0.04}Ti_{0.25}Zr_{0.08}$

Same constituents as data shown with red circles, but different at.% (and fabrication method) produced nearly 2x the strength up to 900 K

(adapted from: Miracle & Senkov, Acta Mat, 201

Conclusions and Future Directions

AM Kustalloy exhibits:

Four distinct phases, with multimodal grain size

Evidence of highly coherent phase boundaries

Exceptional hardness (H ~ 10-15 GPa) and fracture toughness (K_c ~ 70 Mpa-$m^{1/2}$)

Low density ($\rho = 5.7\ g/cm^3$)

Retention of H > 10 GPa up to T = 500 ºC

Moving forward:

Phase identification!

Assessment of ductility – punch tests are in the works (Ames)

Wear testing (SNL)

corrosion testing (SNL)

accelerated aging (oxidation) testing (SNL)

Acknowledging the Many Contributors to this Story

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Nicolas Argibay (Ames), Iver Anderson (Ames), Emma White (Ames/DECHEMA), Duane Johnson (ISU), Prashant Singh (Ames), and Irene Beyerlein (UCSB)

…and probably several others who I inadvertently

Questions
&
Thoughts?