

Tribological and Mechanical Properties of High Entropy Alloys



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2

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Motivation for Metals Tribology Research

cell phones



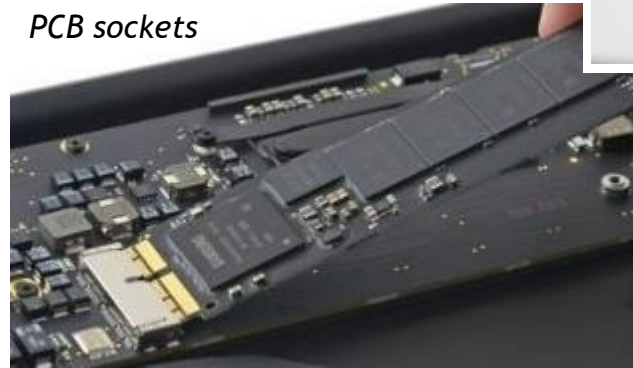
CPU sockets



wind turbine slip-rings
(sensors and blade pitch motors)



PCB sockets



**Estimated 150 Metric Tons (\$6.9B) of Au
used in Electrical Contacts per Year:**

*Refs: Gold Survey, Gold Fields Mineral Survey Ltd, 2011
Gold Bulletin 2010, Vol. 43-3, C. Hagelüken and C.W. Corti,
Gold Bulletin 1986, Vol. 19-3, T.D. Cooke*

45 connectors



EV charging



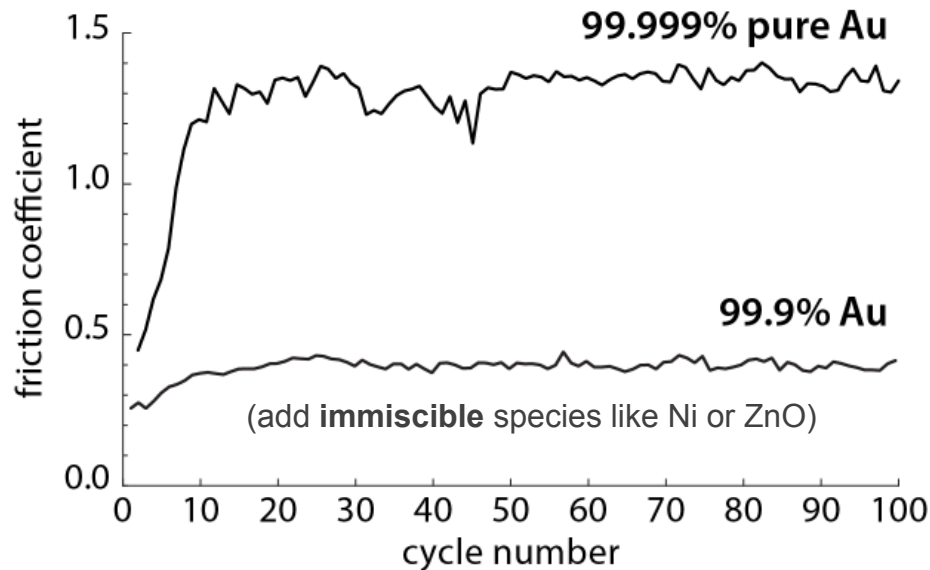
spacecraft slip-rings



Structure-Dependent Friction of (Bare) Metals

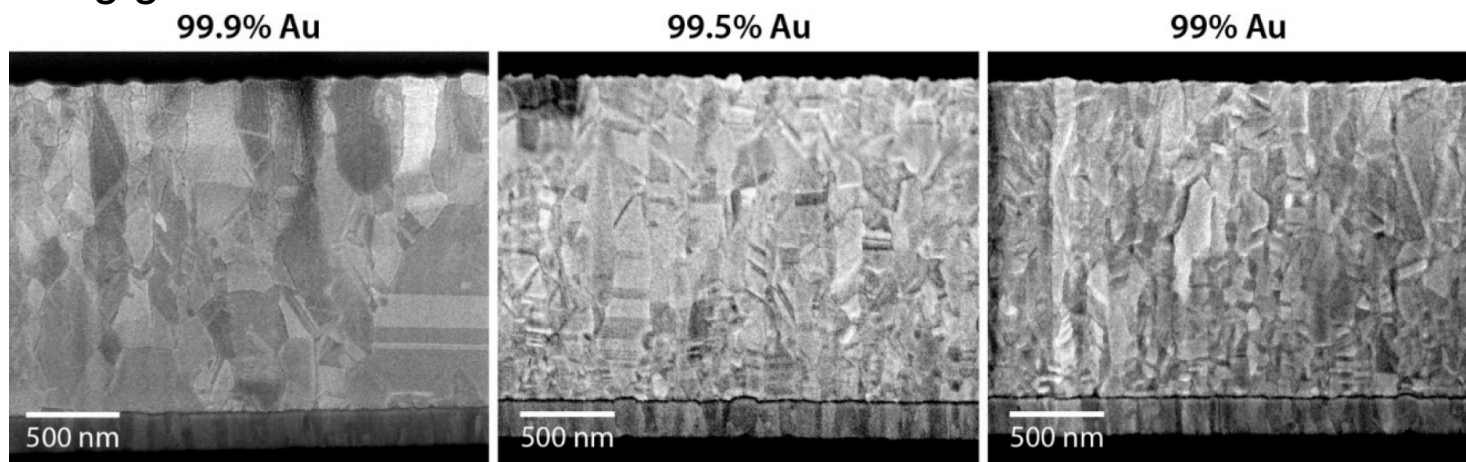


Alloying reduces friction coefficient:



Alloying improves friction & wear performance by reducing and stabilizing grain size

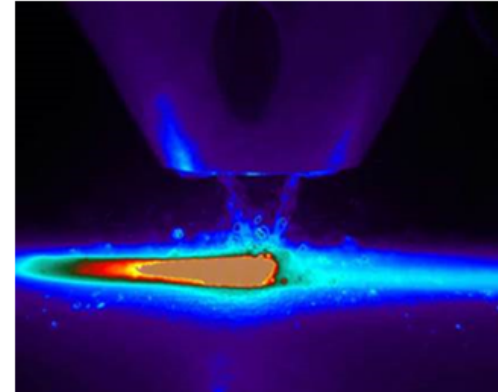
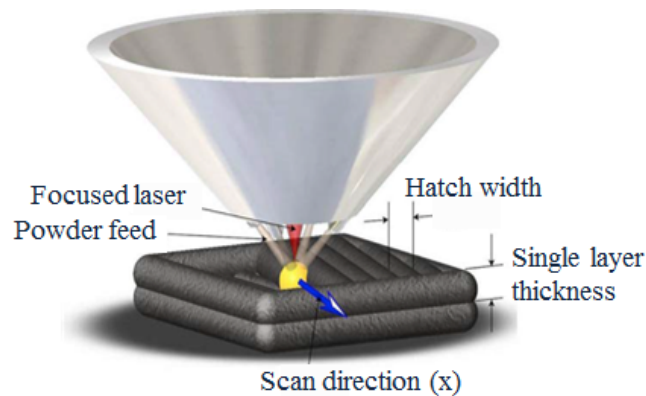
...by reducing grain size:



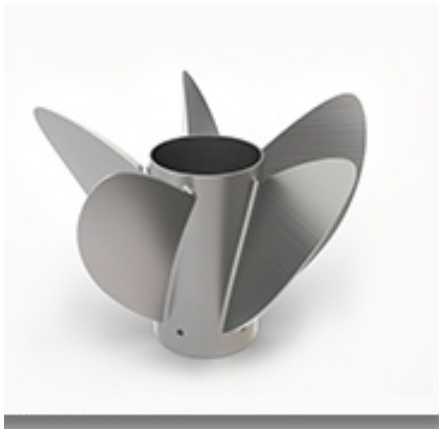
Realizing the Potential of AM



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):

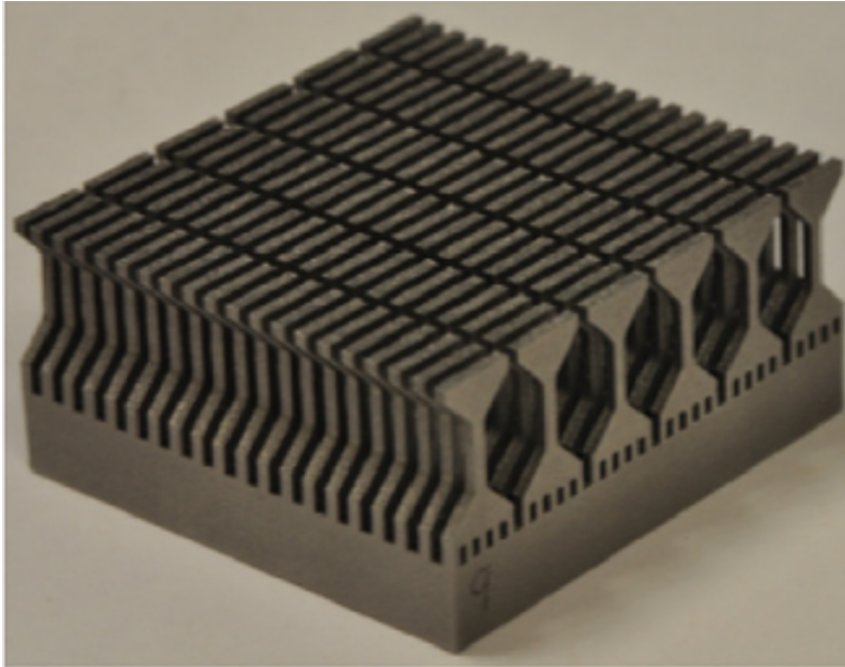


Prototype
Airbus
A380
bracket

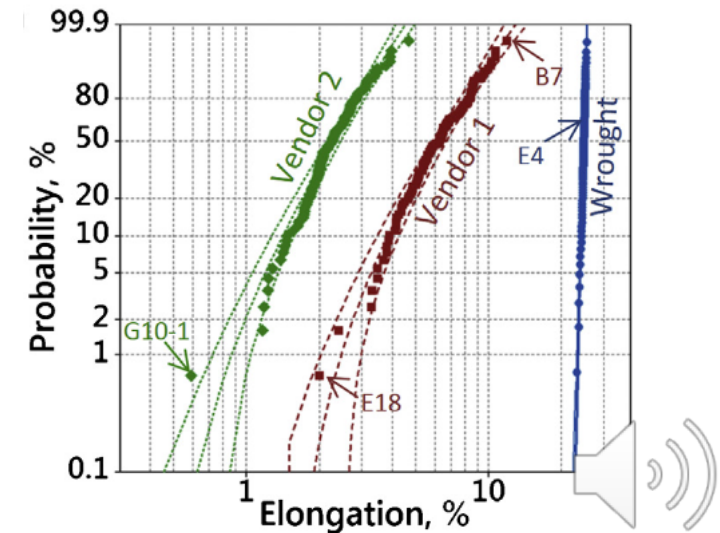
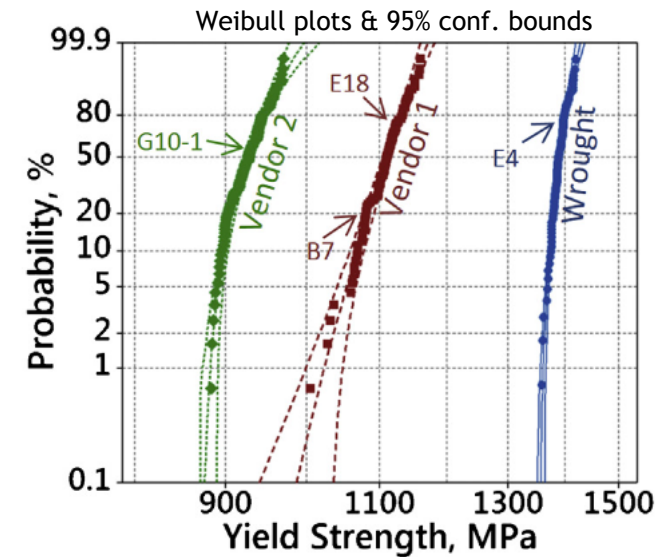
But Conventional Alloys (Steel) Not Optimal For AM



Laser consolidated (powder bed) 17-4 PH Steel:



Conclusion: Reduced strength AND elongation to failure (ductility) attributed to higher surface roughness, porosity, and microstructure (e.g., grain size).



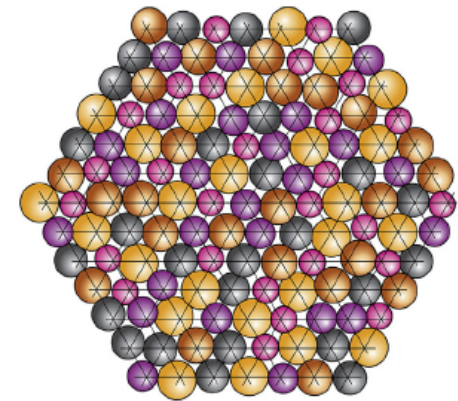
Why Are We Printing High-Entropy Alloys?



High Entropy Alloys: primarily solid solution alloys that contain 5+ alloying constituents, where microstructures have high configurational entropy ($\geq 1.4R$).

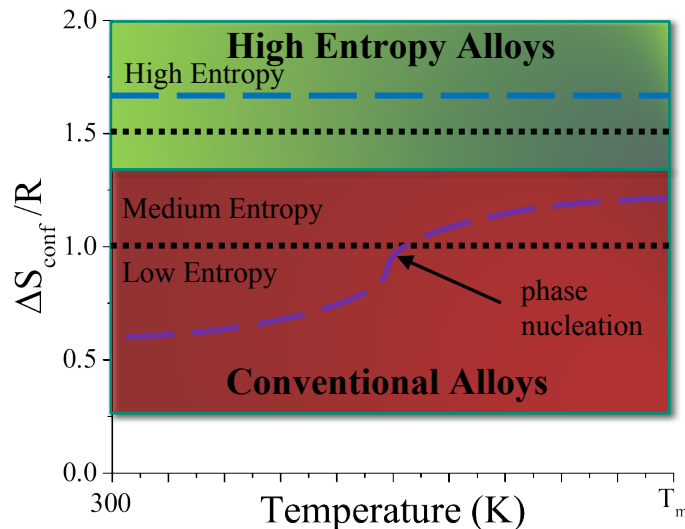
Competition between Gibbs energy for solid solution and intermetallic formation

$$\Delta G^{SS} < \Delta G^{IM} \rightarrow \Delta S^{SS} > \frac{\Delta H^{IM} - \Delta H^{SS}}{T}$$



disordered solid solutions

Miracle, et al., Acta Mat., 2017

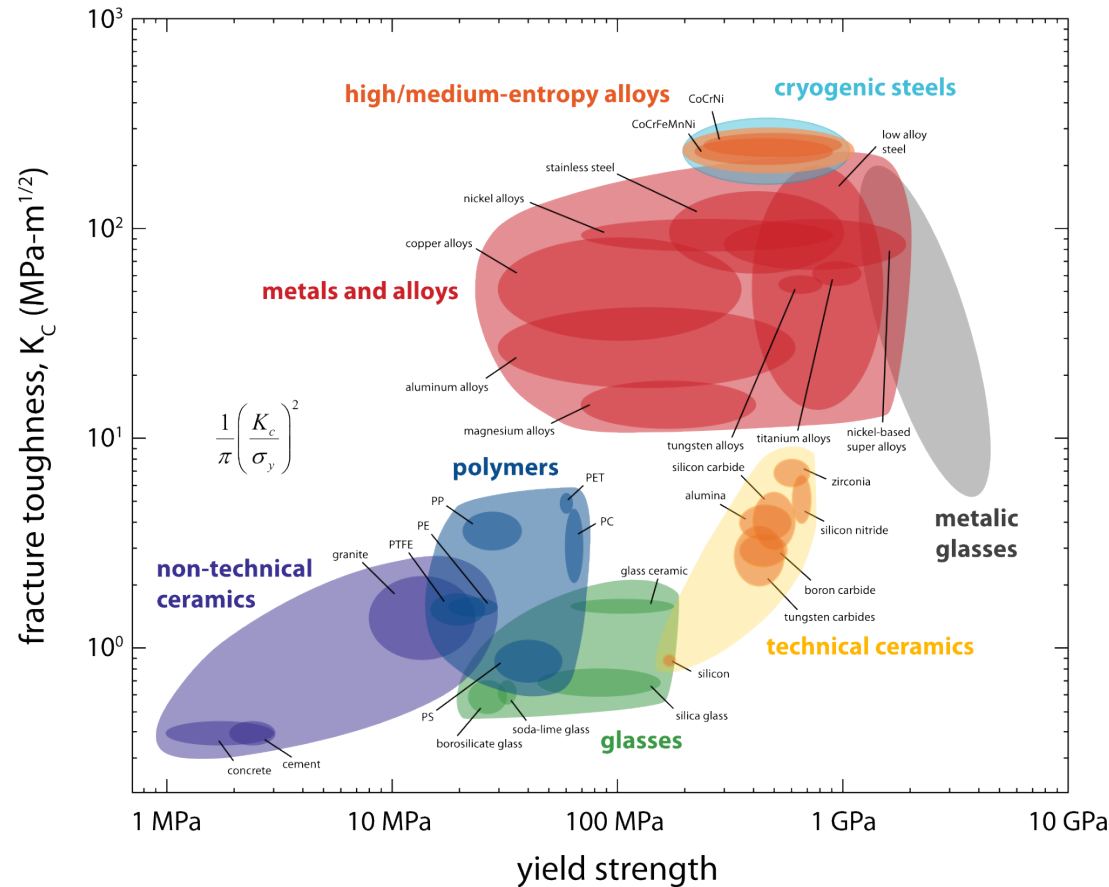


D. Miracle et al., Entropy, 2014

These alloys derive much of their strength from solution strengthening, and are **insensitive to processing conditions (i.e., thermal history)**.



Why Are We Printing High-Entropy Alloys?

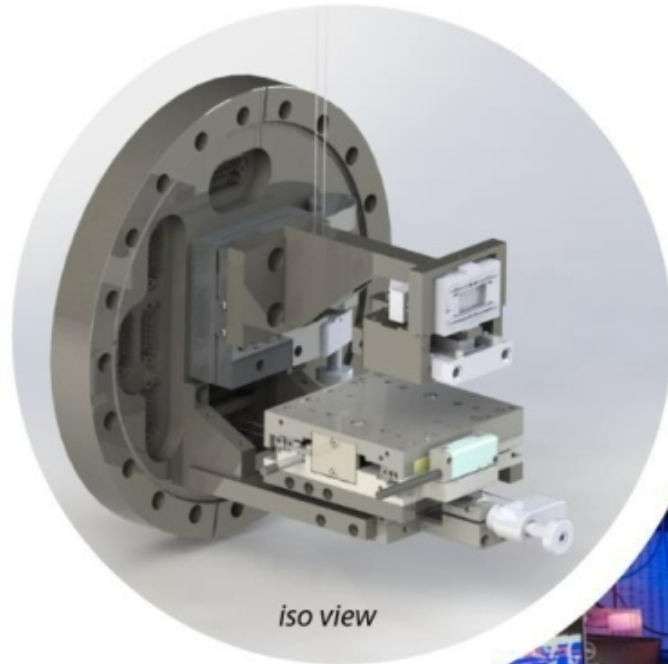


HEAs have properties exceeding most conventional alloys, suggesting improved resistance to failures AM defects.

Goal: demonstrate these alloys as a materials-based solution to *achieve the promise of metal AM, i.e. insertion into high consequence applications.*



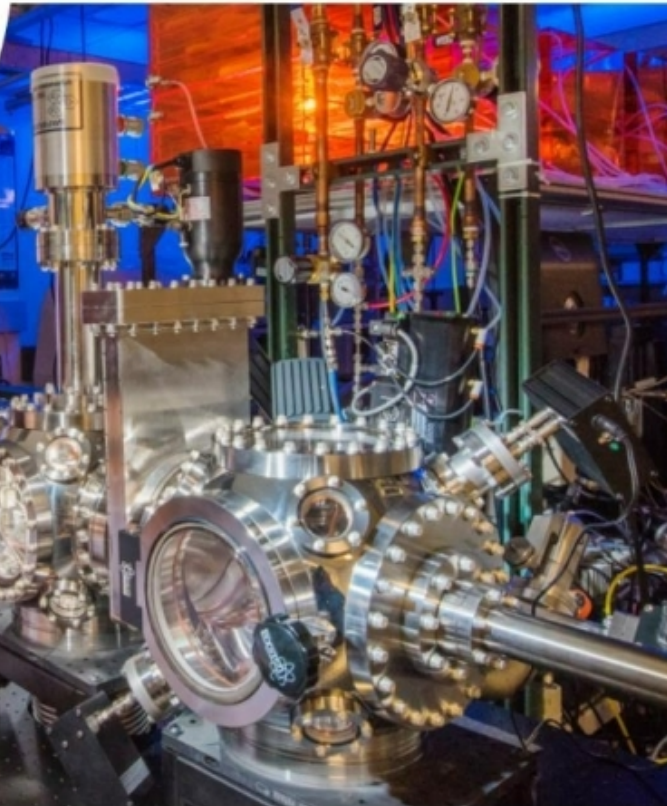
A Starting Point: Use UHV tribometer to Probe (Bare) Metal



iso view

- High fidelity, capacitive displacement & flexural cantilever based system situated on 8" CF vacuum flange

- Two chambers: allows for fast entry transfer system or a second tribometer

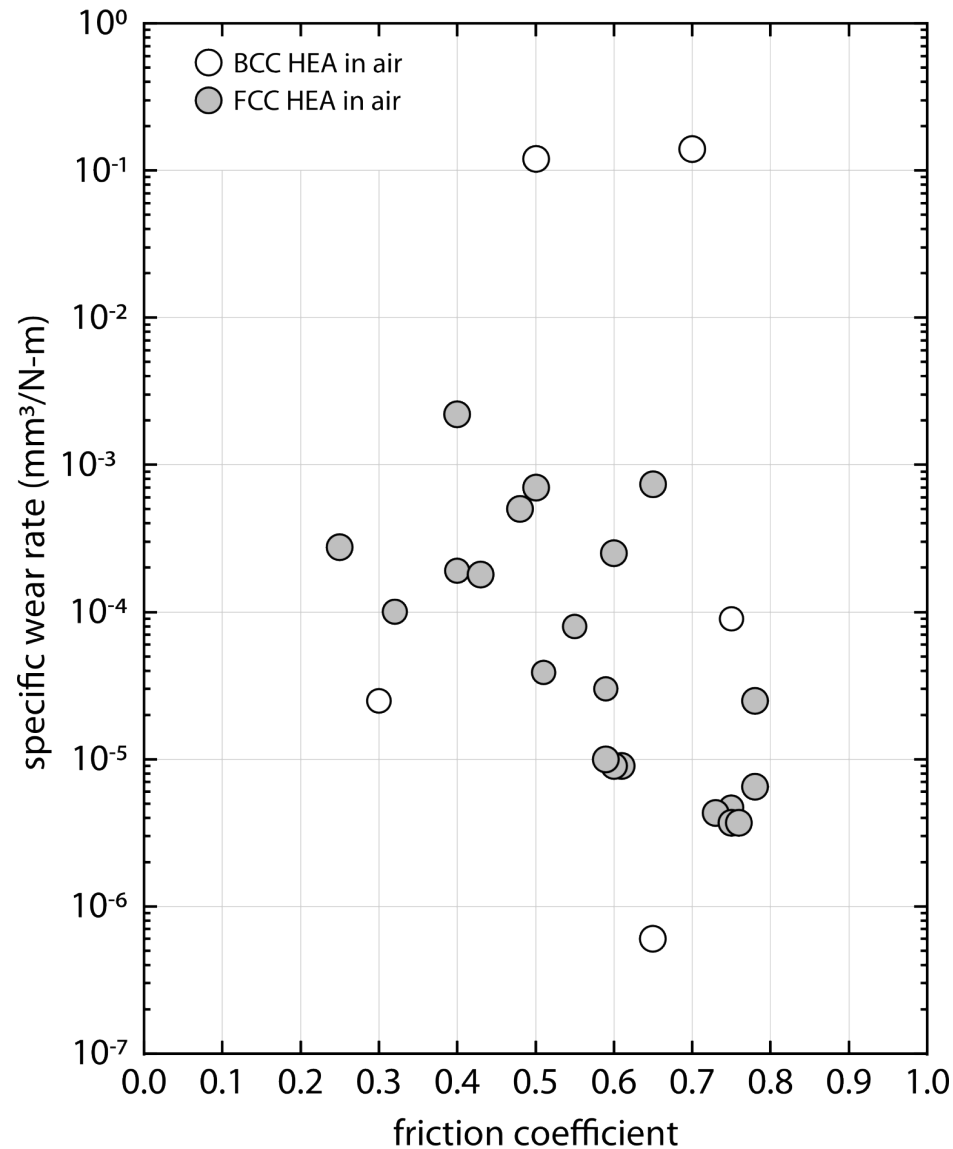


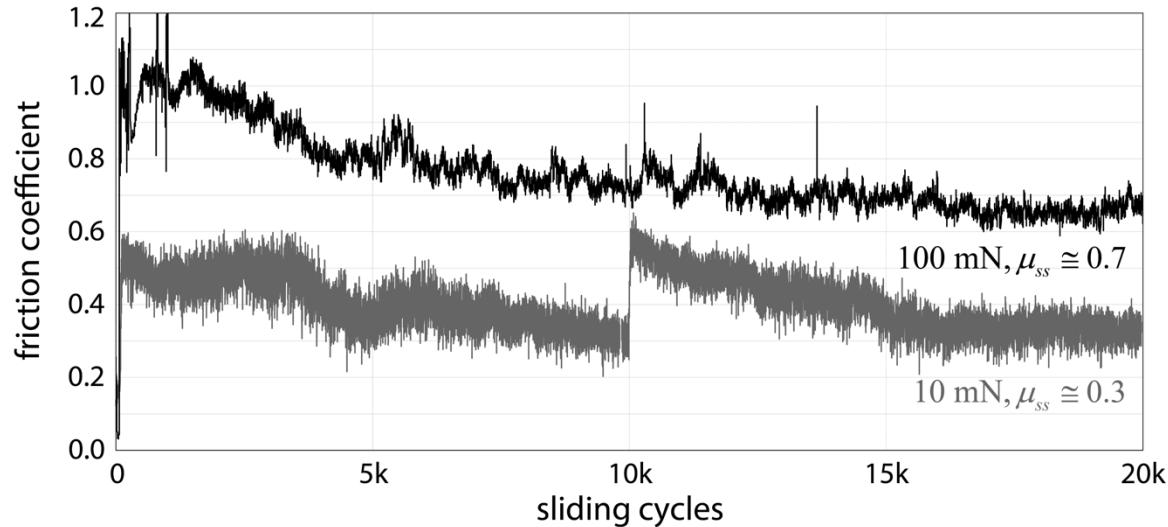
Specs

- Linear reciprocating motion
- Load metering (0.1 mN to 1N)
- Cryo stage (4–800K; actual +/- 150C)
- 10^0 to 10^{-9} torr
- 100 $\mu\text{m/s}$ – 100 mm/s
- 10 kHz acquisition

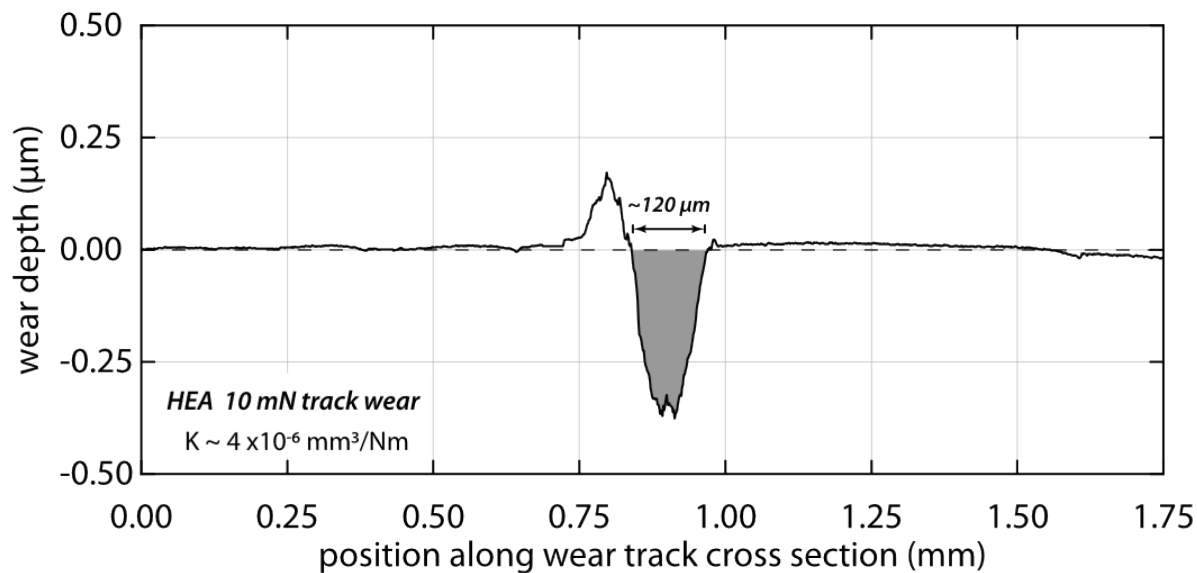


Literature Survey ... No Data for Bare Metal Properties

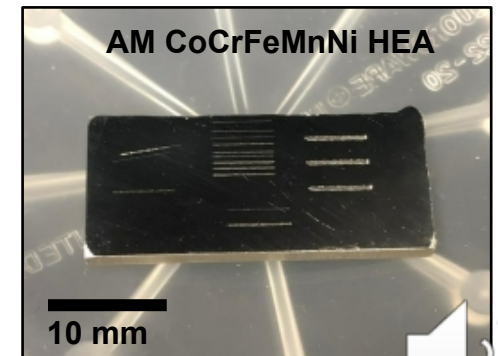




UHV testing in the absence of environment allows us to probe fundamental friction behavior of Cantor HEA sliding against a sapphire sphere under different loading conditions.



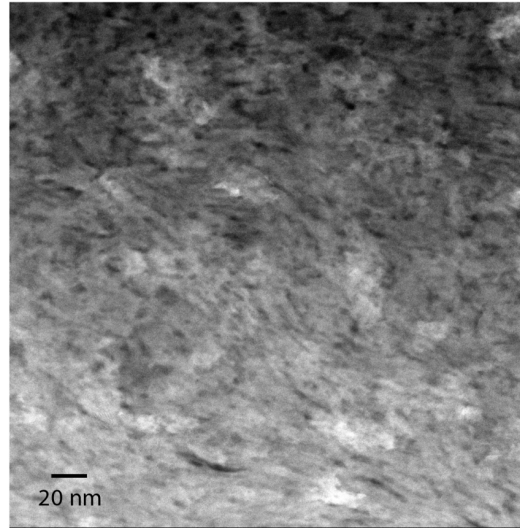
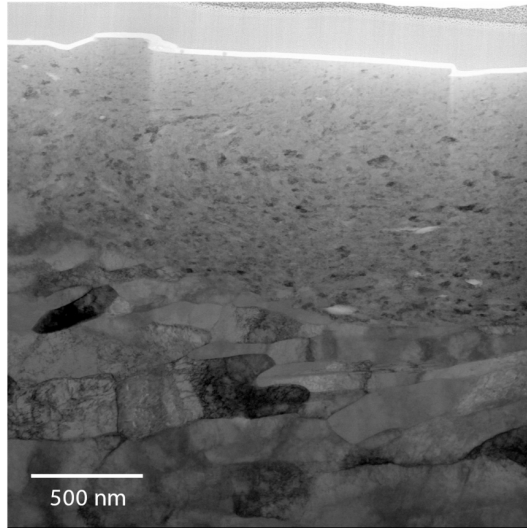
For a relatively soft material, it exhibited high wear resistance.



Different Friction Behavior - TEM of Wear Track Microstructure



Representative cross-section TEM of $\mu = 0.32$ wear track

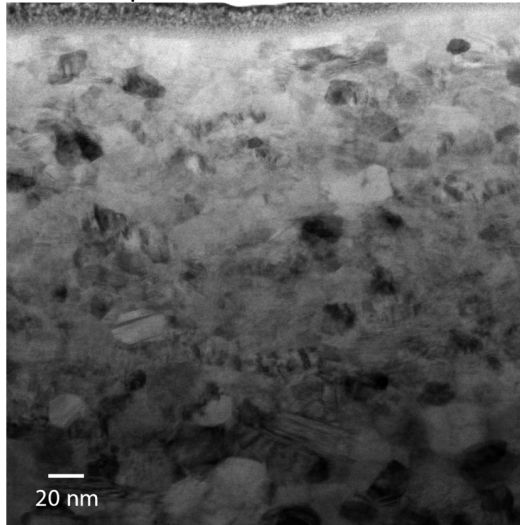
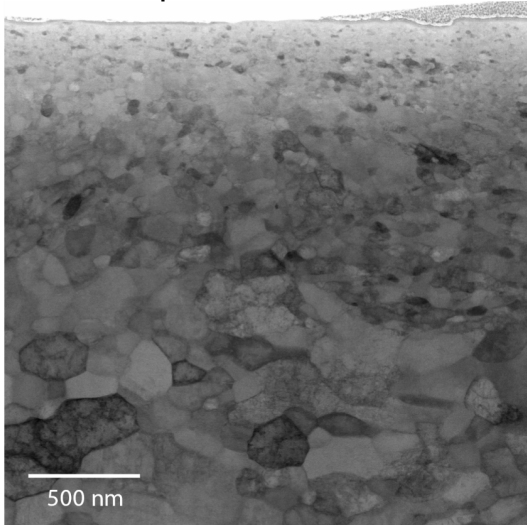


initial grain size 1-10 μm

10 mN track showed an average grain size below 10 nm

100 mN track showed a slightly larger average grain size of ~ 20 nm

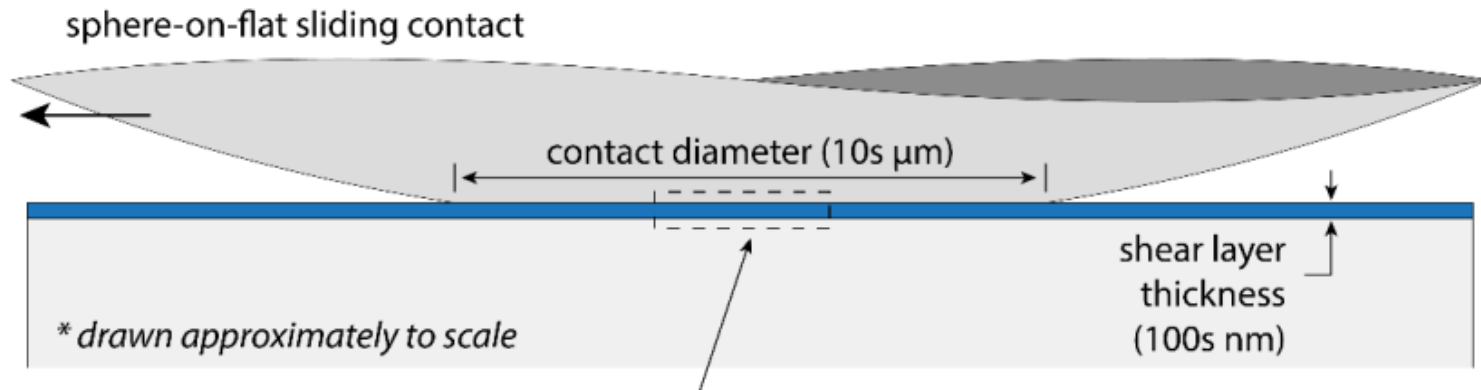
Representative cross-section TEM of $\mu = 0.68$ wear track



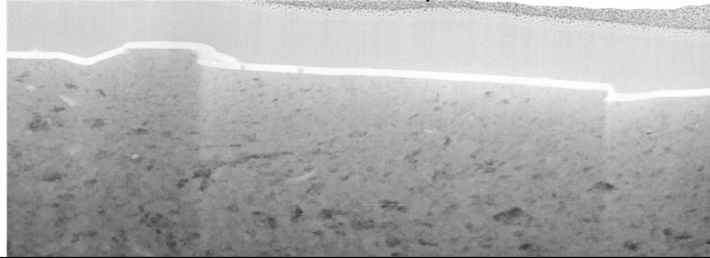
low friction 10 mN wear track surface falls within inverse HP regime



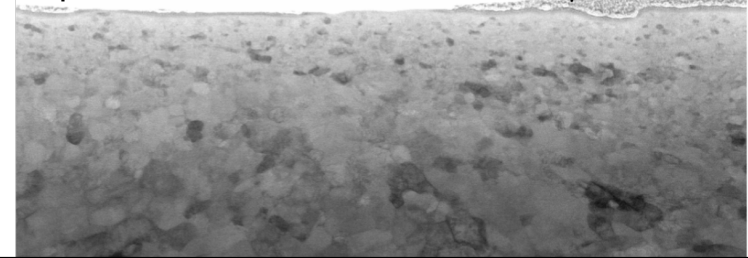
Lower Friction is a Result of Surface Grain Refinement



Representative cross-section TEM of $\mu = 0.32$ wear track



Representative cross-section TEM of $\mu = 0.68$ wear track



It is useful to define the friction coefficient of metals as the ratio of interface shear strength and bulk hardness:

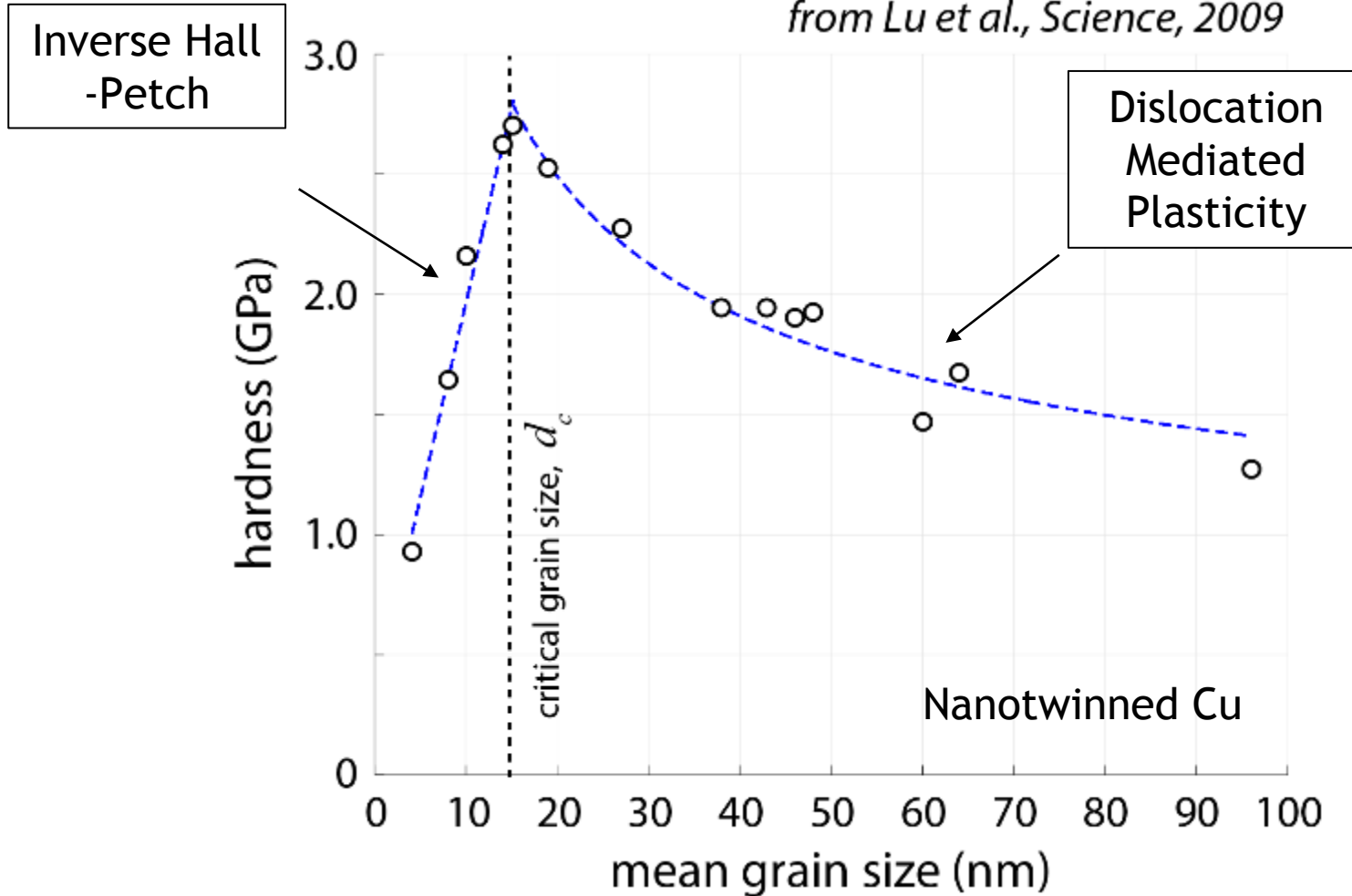
Bowden & Tabor: $\mu = \frac{\tau}{H}$ $\xrightarrow{\text{Tabor}}$ $\mu = \frac{\tau}{3 \cdot \sigma_Y}$ $\xrightarrow{\text{von Mises}}$ $\mu = \frac{\tau(d_{\text{surface}})}{3\sqrt{3} \cdot \tau(d_{\text{bulk}})}$

500 nm

500 nm



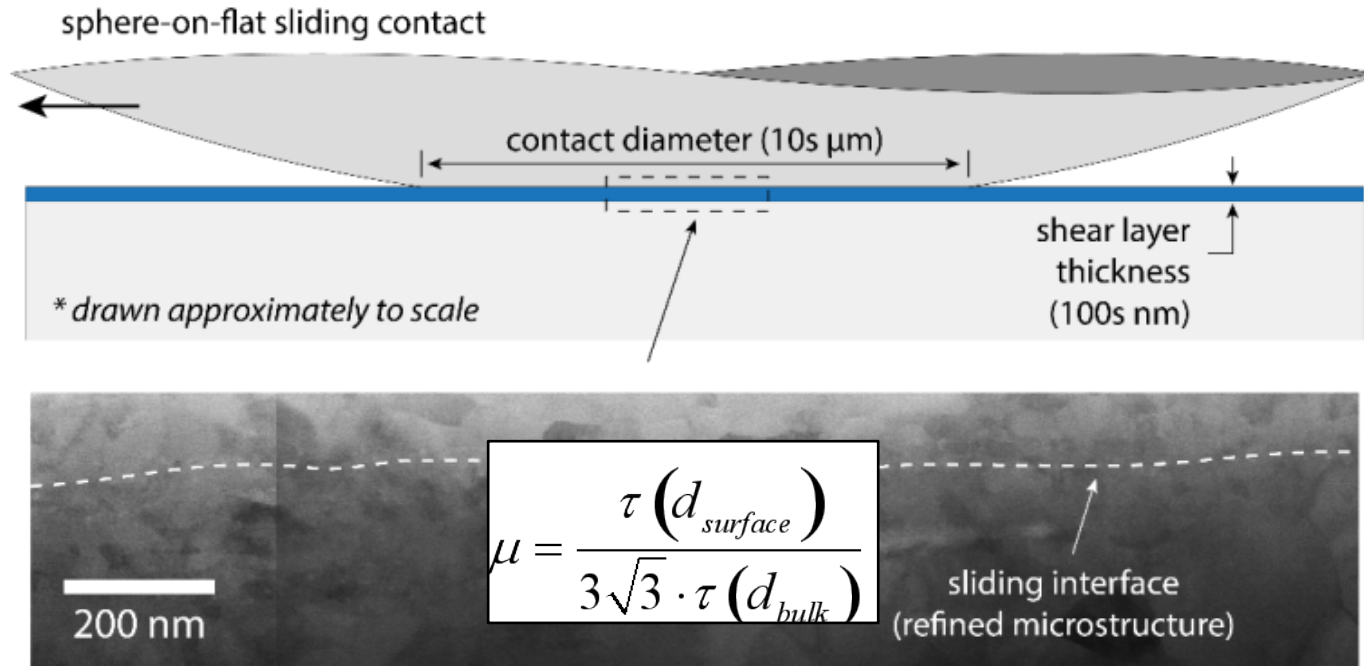
Inverse Hall-Petch Regime (Softening) is Key to Low Friction



Fundamental mechanism of softening during sliding remained poorly understood...



New Model Developed Based on Dynamic Amorphization



Energy barrier (or stress) for the creation of a dynamic amorphous interface, which is analogous to generating a continuous high angle grain boundary...

Amorphization model:

$$\tau(d) = \left(L \frac{\rho_L}{M} \right) \left(1 - \frac{T}{T_m} \right) \left(\frac{d - \delta}{d} \right)^3 + \left(\frac{\dot{\epsilon}_{GBS}}{\dot{\epsilon}_{tot}} - 1 \right) \frac{kT}{b^3}$$

atomic volume of amorphous metal

volume fraction of GB to crystal

thermal energy

heat of fusion

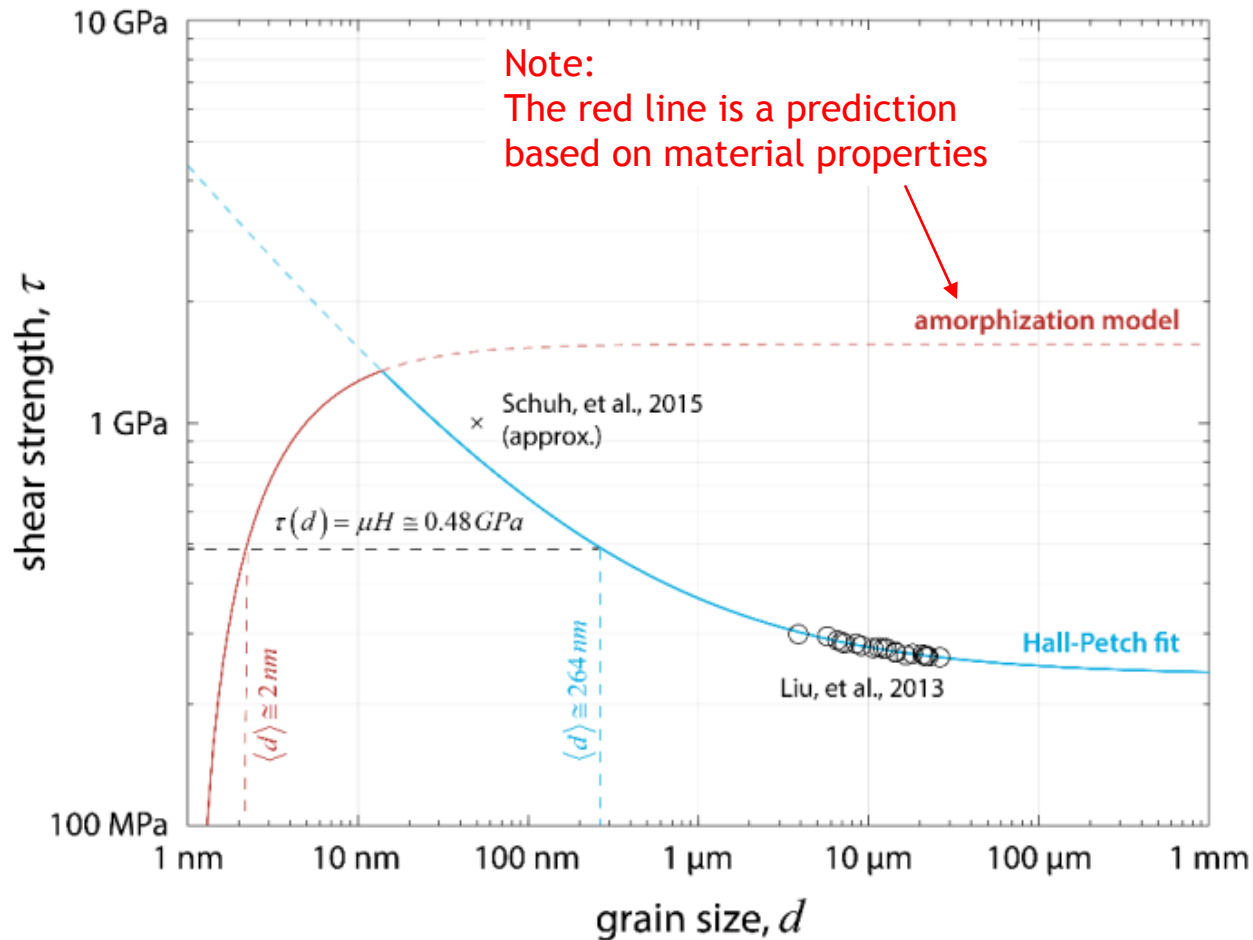
homologous temperature

strain rate (0 to 1)

per atomic diameter³

No fitting parameters!



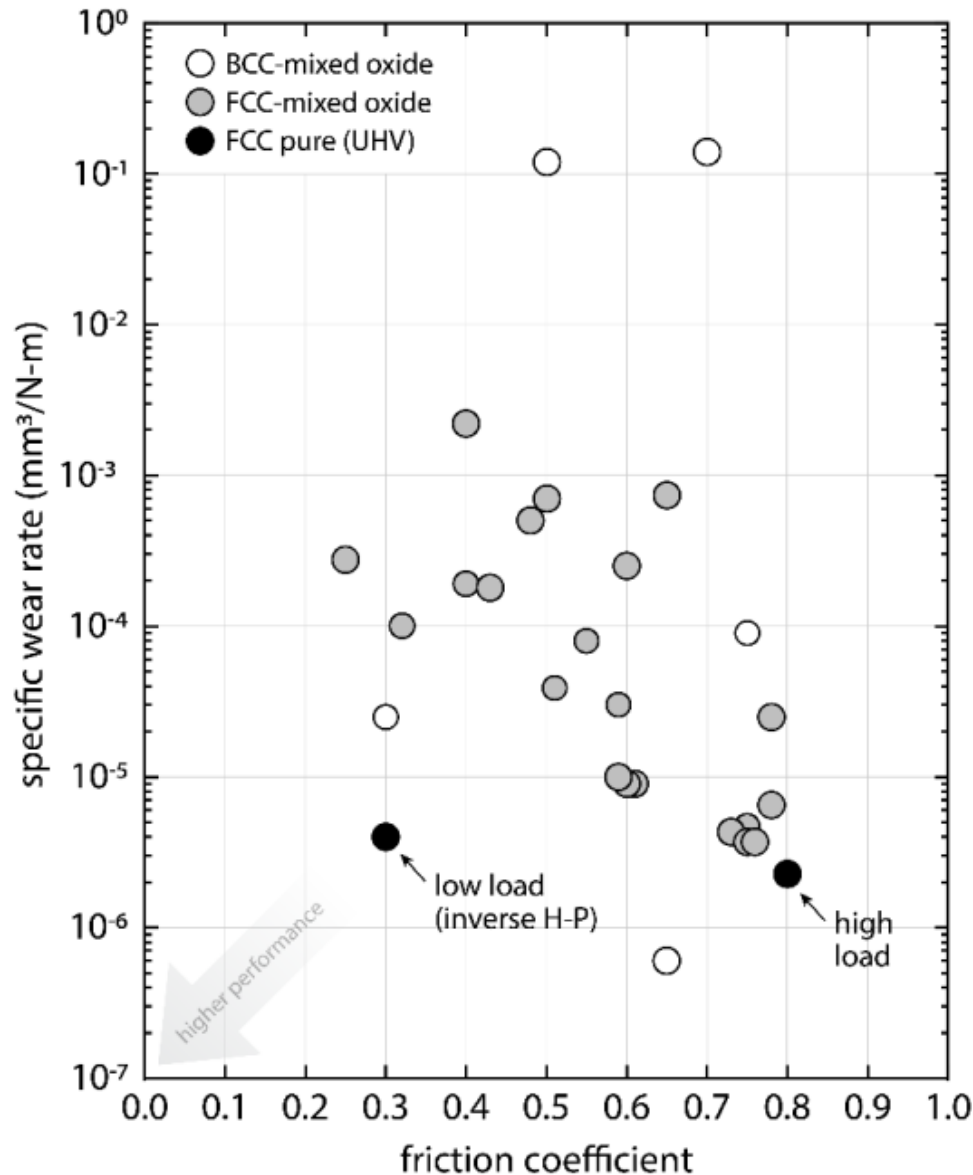


Bulk hardness = 1.6 GPa, so for:

$$\mu = 0.3 \rightarrow \tau(d_{\text{surface}}) = \mu H \cong 0.48 \text{ GPa}$$

$$\mu = 0.7 \rightarrow \tau(d_{\text{surface}}) = \mu H \cong 1.12 \text{ GPa}$$



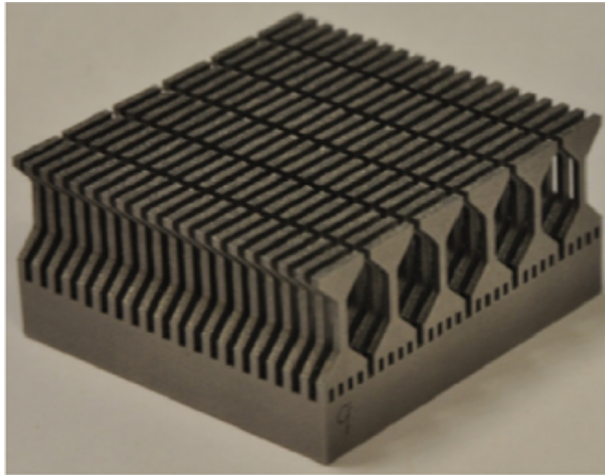


mixed metal-oxide HEAs, as do pure metals, exhibit very different properties than the metal without oxidation

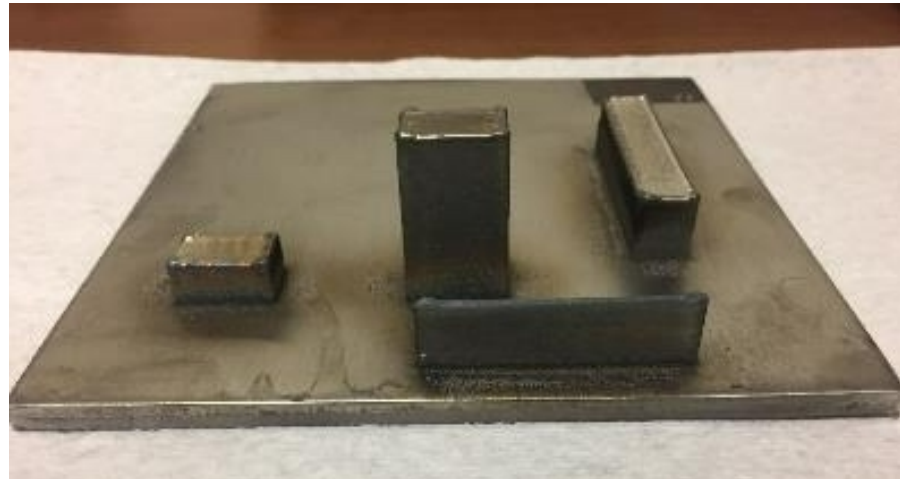
under both a 10 mN and 100 mN normal load, the AM Cantor HEA exhibited high wear resistance



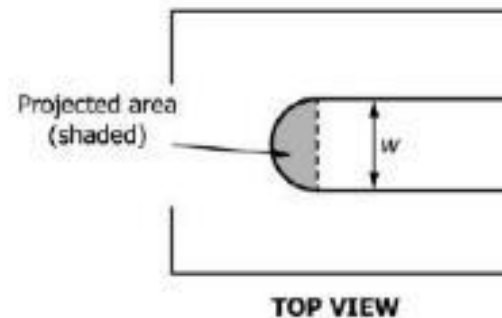
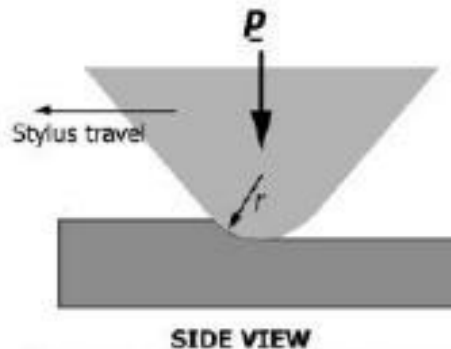
AM+ Tribology – Rapid Materials Discovery (Synthesis and Characterization)



Challenging to Make
(Inconsistent because of defects)



Easy to Make
(Ideal for Tribological Testing)



NOTE 1—The contact of the stylus is assumed to produce a semi-circular projected area when viewed from the top.

FIG. 2 Final Scratch Width



AM+ Tribology – Rapid Materials Discovery (Synthesis and Characterization)



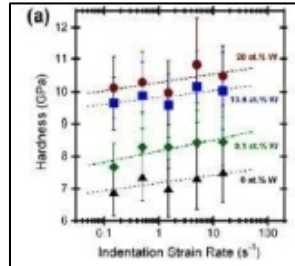
Enhanced solid solution effects on the strength of nanocrystalline alloys

Timothy J. Rupert, Jonathan C. Trenkle, Christopher A. Schuh*

Department of Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA 02139, USA

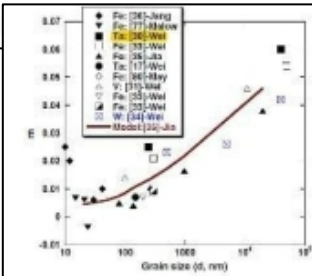
Received 17 October 2003; revised in final form 8 November 2003; accepted 9 November 2003

Available online 8 December 2003



Strain rate effects in the ultrafine grain and nanocrystalline regimes—influence on some constitutive responses

Q. Wei



STRAIN HARDENING OF SINGLE ALUMINUM CRYSTALS DURING POLYSLIP

A. K. Mukherjee,¹ J. D. Mote² and J. E. Dorn³

The temperature and strain rate dependence of the flow stress of tantalum

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Energy Research and Development Administration, Washington, D.C., USA

A. K. MUKHERJEE

Department of Mechanical Engineering and Materials and Devices Research Group, University of California, Davis, California, USA

A MICRO-INDENTATION STUDY OF SUPERPLASTICITY IN Pb, Sn, AND Sn-38 wt% Pb

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Department of Materials Science and Engineering, Stanford University, Stanford, CA 94305, U.S.A.

(Received 7 December 1987)

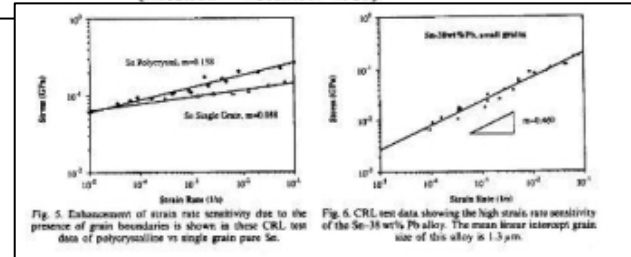


Fig. 5. Enhancement of strain rate sensitivity due to the presence of grain boundaries is shown in these CRL test data of polycrystalline vs. single grain pure Sn.

Fig. 6. CRL test data showing the high strain rate sensitivity of the Sn-38 wt% Pb alloy. The mean linear intercept grain size of this alloy is 1.5 μm .

Characterization of Strain-Rate Sensitivity and Grain Boundary Structure in Nanocrystalline Gold-Copper Alloys

LUKE O. NYAKITI and ALAN F. JANKOWSKI

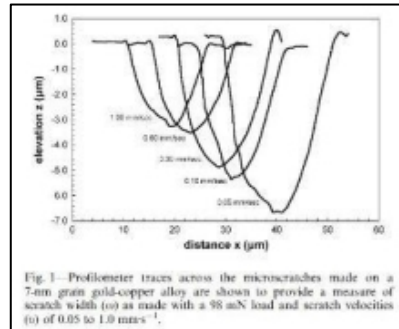


Fig. 1. Profilometer traces across the microcracks made on a 7-nm grain gold-copper alloy are shown to provide a measure of scratch width (a) as made with a 98 mN load and scratch velocities (b) of 0.05 to 1.0 mm s⁻¹.

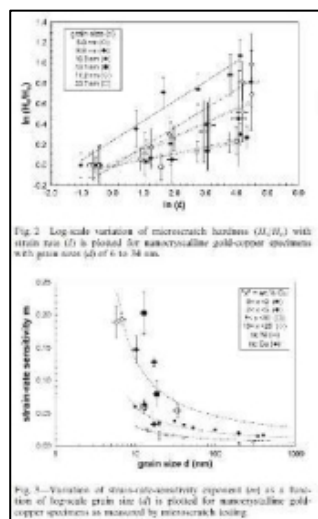


Fig. 2. Logarithmic variation of microscratch hardness (H_0/H_a) with strain rate ($\dot{\gamma}$) is plotted for nanocrystalline gold-copper specimens with grain sizes (d) of 6 to 34 nm.

Fig. 3. Variation of strain-rate sensitivity exponent (n) as a function of logarithmic grain size (d) is plotted for nanocrystalline gold-copper specimens as measured by microscratch testing.

Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology

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The University of Tennessee at Oak Ridge National Laboratory, Department of Materials Science and Engineering, Eastville, Tennessee, 37830

Standard Test Method for Scratch Hardness of Materials Using a Diamond Stylus¹

This standard is issued under the fixed designation G171; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.



NOTE 1—The contact of the stylus is assumed to produce a semi-circular projected area when viewed from the top.

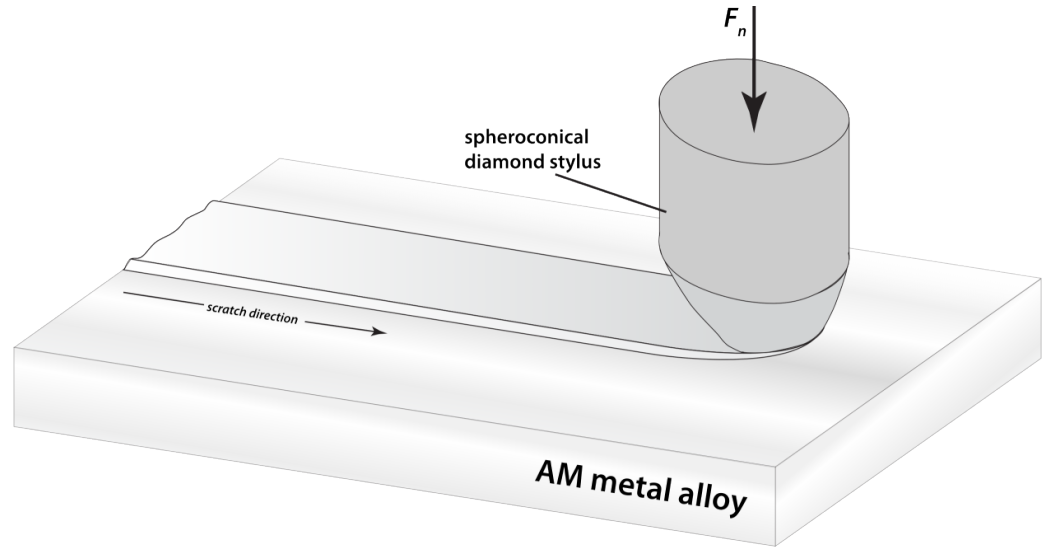
FIG. 2 Final Scratch Width

High-Throughput Scratch Testing for Rapid Material Characterization



strain rate
(1/s)

hardness
(GPa)



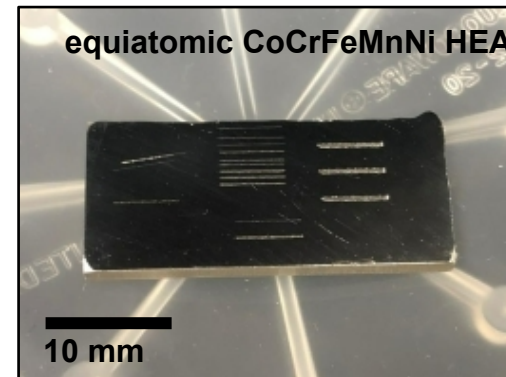
strain rate sensitivity
exponent

activation
volume

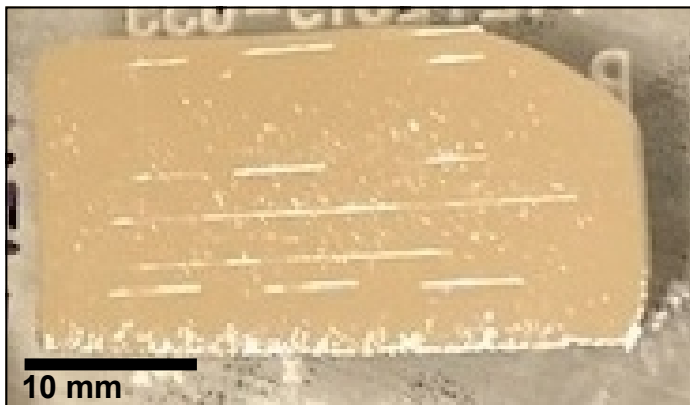
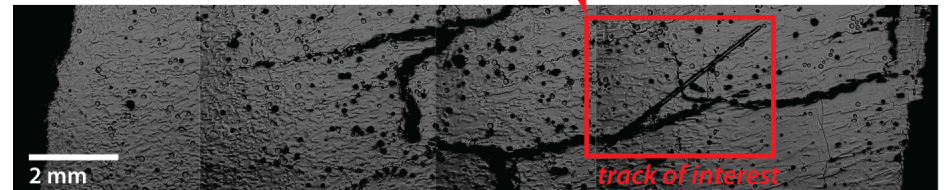
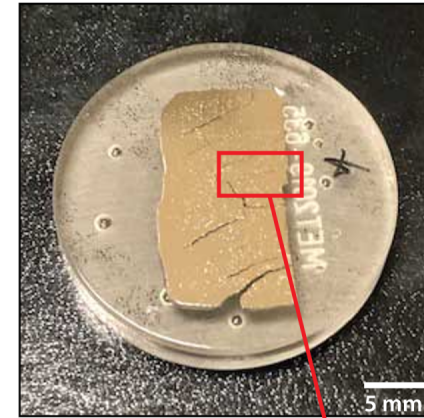
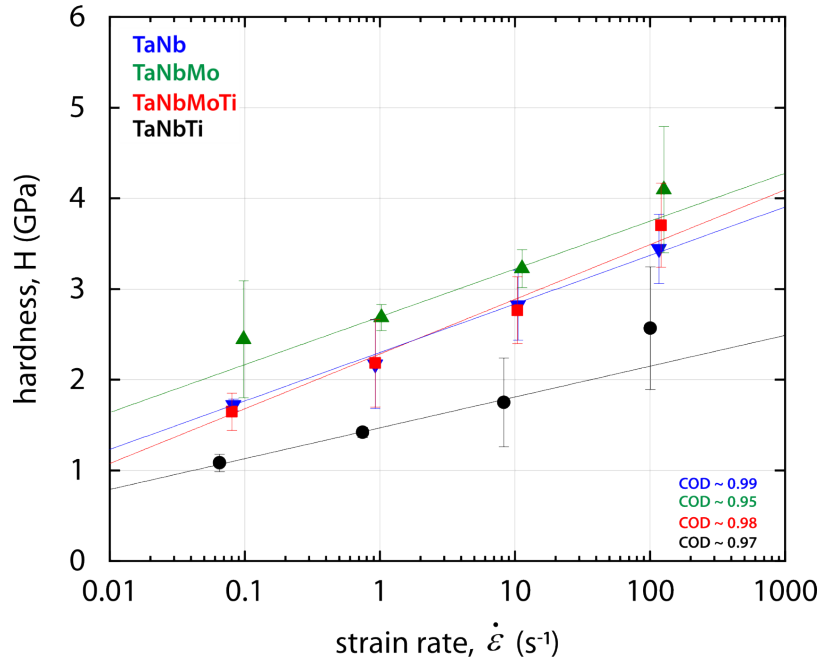
$\propto \sqrt{\quad}$

characteristic
length

$\sqrt{\quad}$



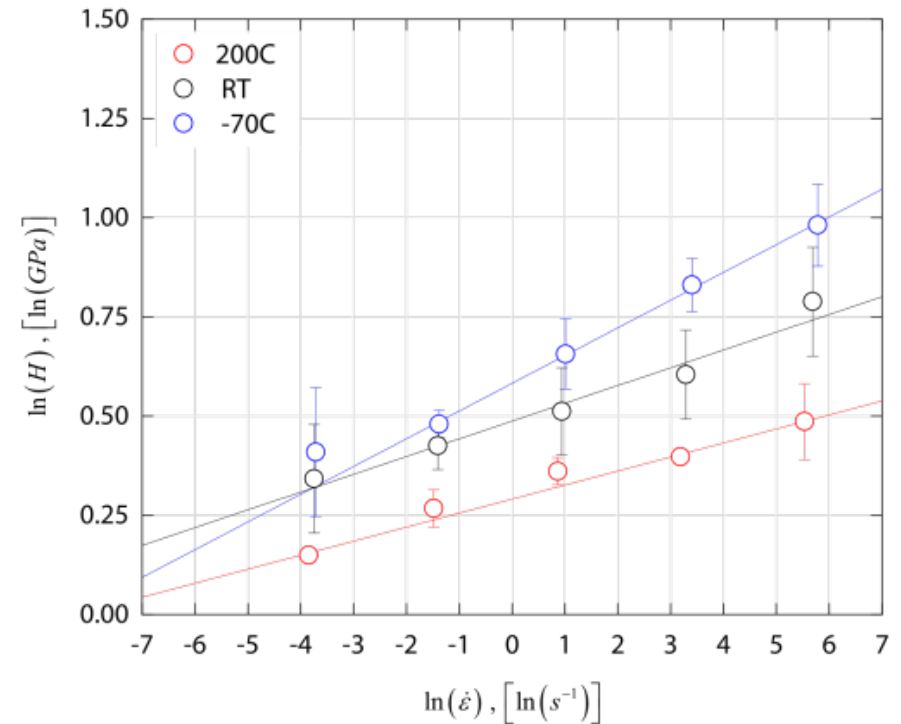
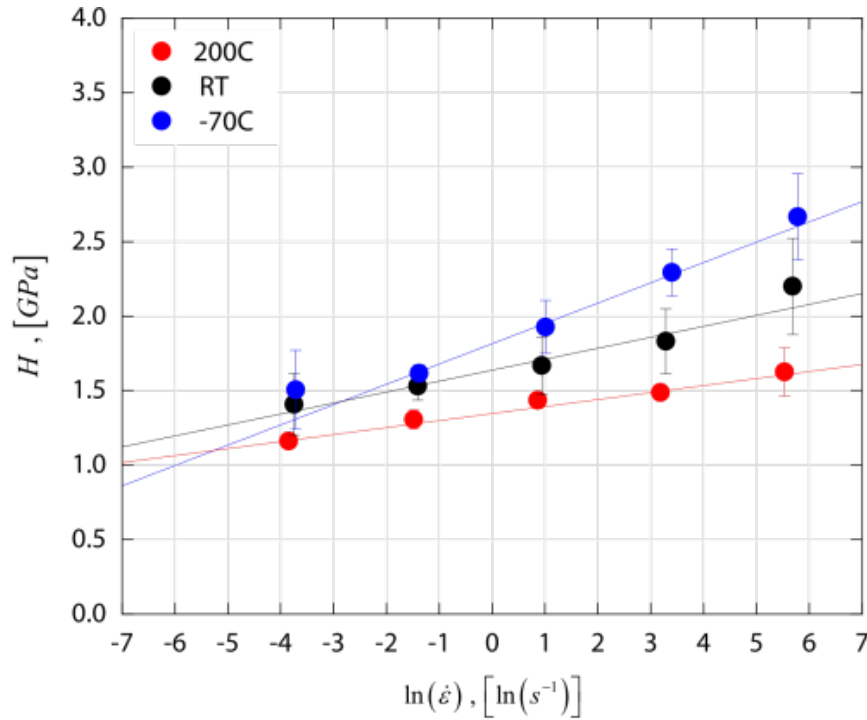
Characterization Possible Even with 'Bad' AM Specimens



Voids, dimples, cracks would make these specimens unusable for tensile testing, but all we need is a polished surface.



AM Cantor HEA Revisited – Temperature and Strain Rate Dependent Hardness



	m	$\sigma(m)$	V (nm ³)	$V^{1/3}$ (nm)	$\sigma(V)$ (nm ³)
CoCrFeMnNi -70C	0.070	0.004	0.158	0.541	0.013
CoCrFeMnNi 25C	0.045	0.005	0.293	0.664	0.035
CoCrFeMnNi 200C	0.035	0.003	0.458	0.771	0.025





- Low friction and wear of metal sliding contacts can be achieved by mitigating thermomechanically-driven microstructural evolution (e.g., grain growth).
- Fundamental investigations of metal contacts led to the development of a simple model that can be used to guide alloy design – prediction of ultimate strength based on materials properties like heat of fusion.
- Evidence that high entropy alloys (HEAs) may be an ideal alloy system for additive manufacturing of tribological and structural components.
- Able to rapidly elucidate hardness, strain rate dependence, fracture toughness, etc. from scratch testing AM specimens.

Questions?

