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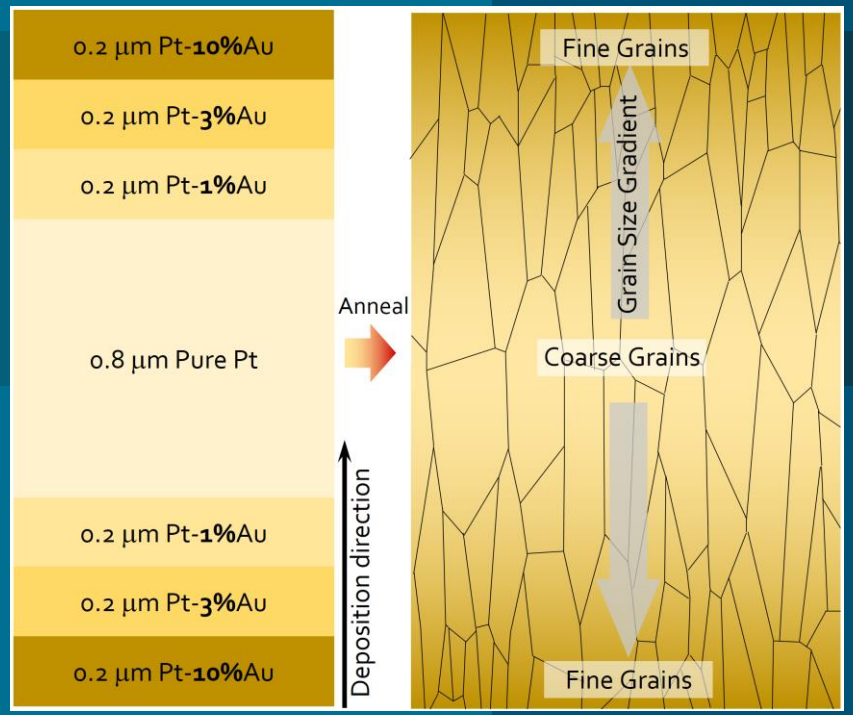
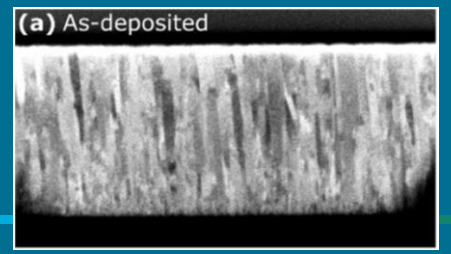
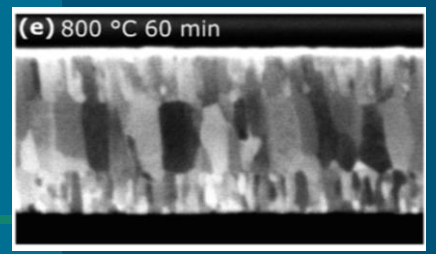
# Gradient Nanostructured Metals via Compositional Means

Alejandro Barrios, James Nathaniel II; Joseph Monti; Khalid Hattar; Douglas Medlin; Remi Dingreville; Brad Boyce

Sandia National Laboratories




TMS 2023 : Deformation Mechanisms, Microstructure Evolution, and Mechanical Properties of Nanoscale Materials

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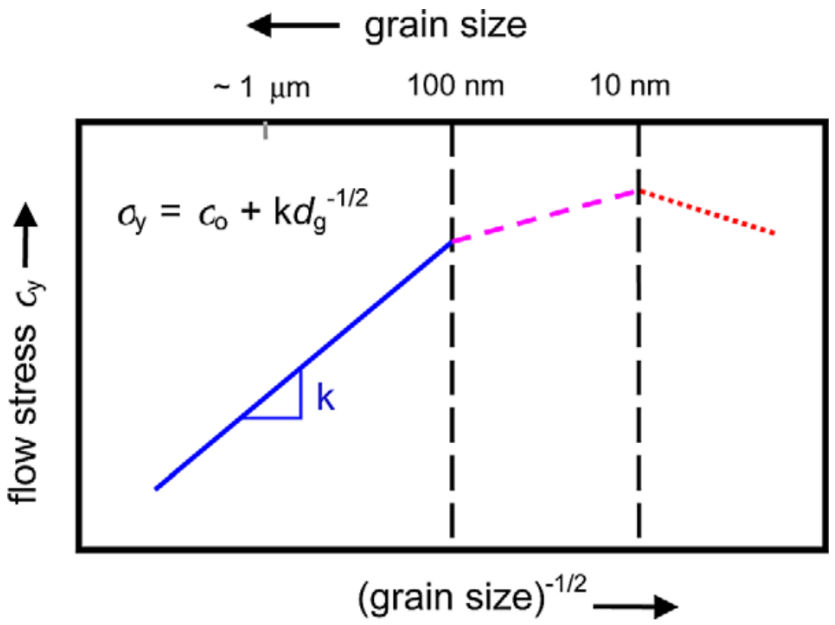
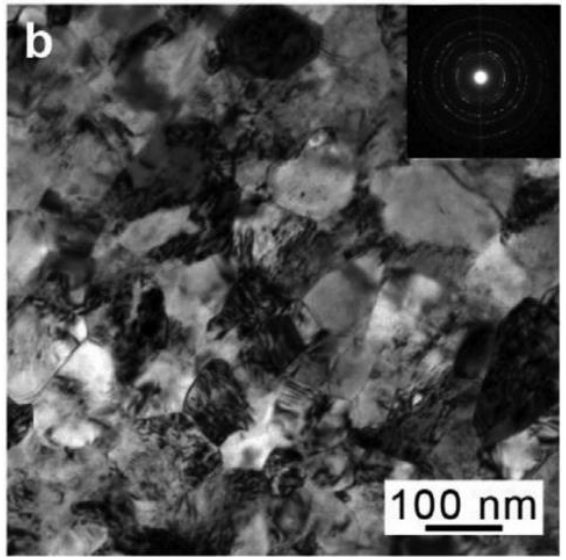
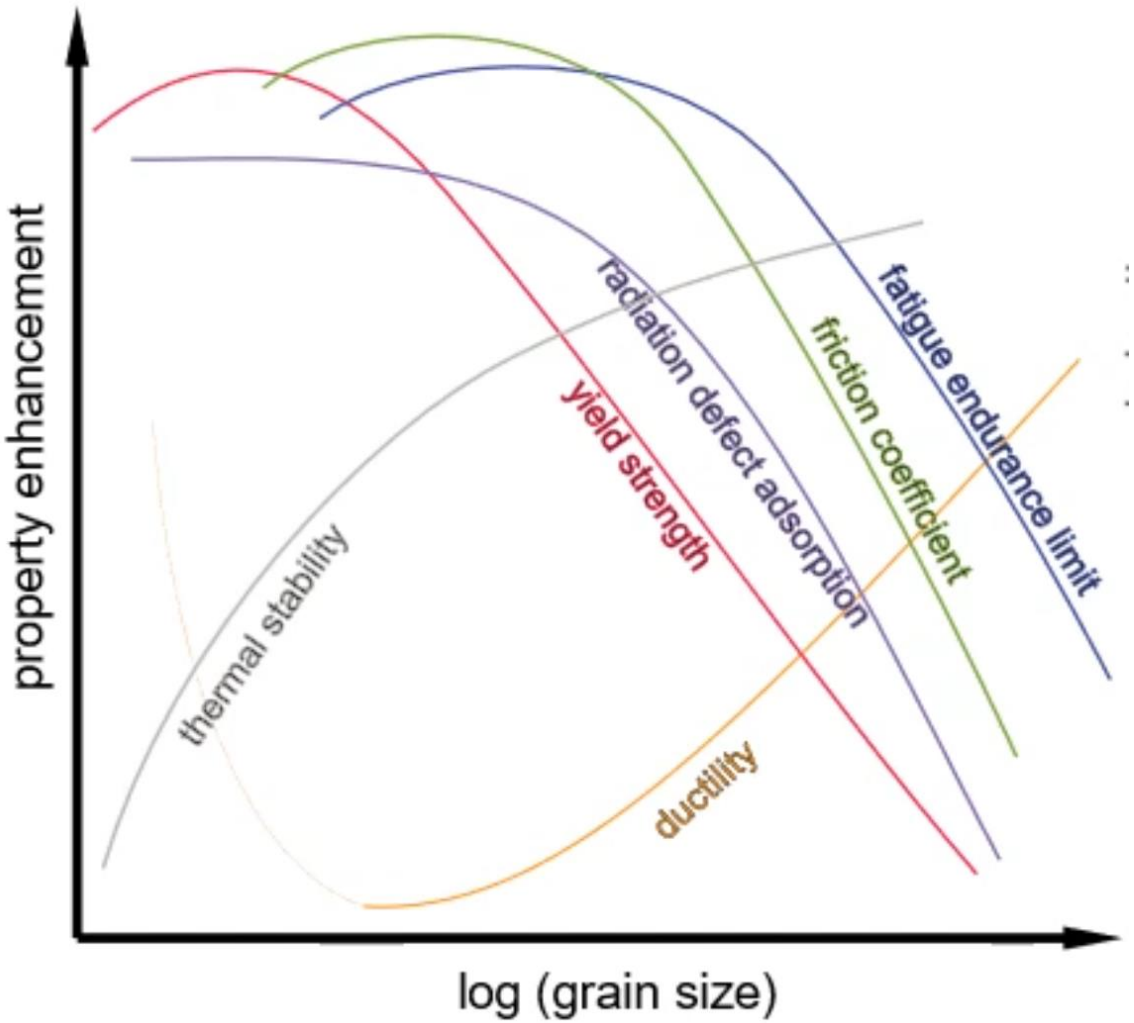
# Introduction: Nanocrystalline Metals



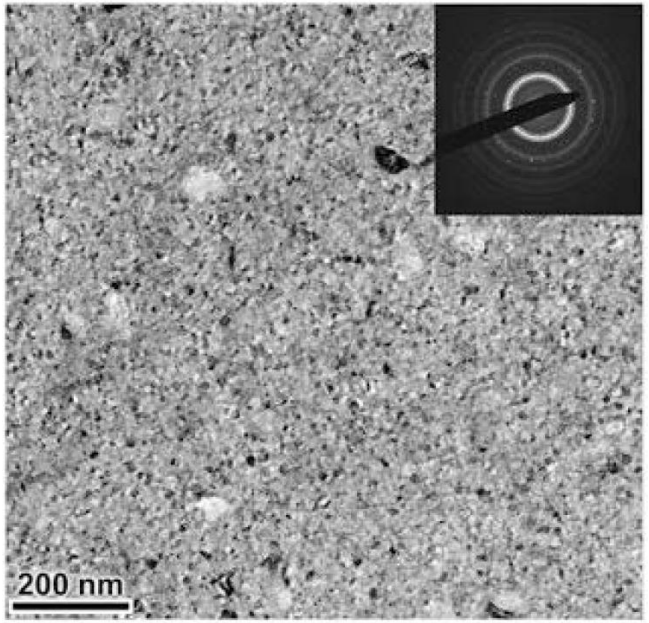



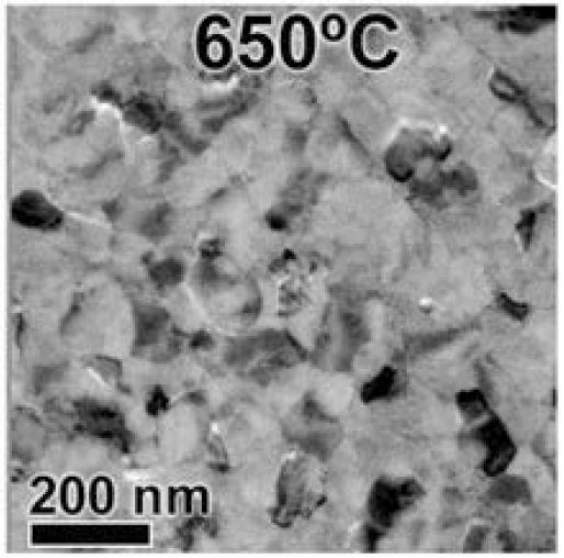

[Long et al., *Acta Mater.*, 2019] [Kumar et al., *Acta Mater.*, 2003]

# Background: Nanocrystalline Metals – Stability and Ductility

*Stability*



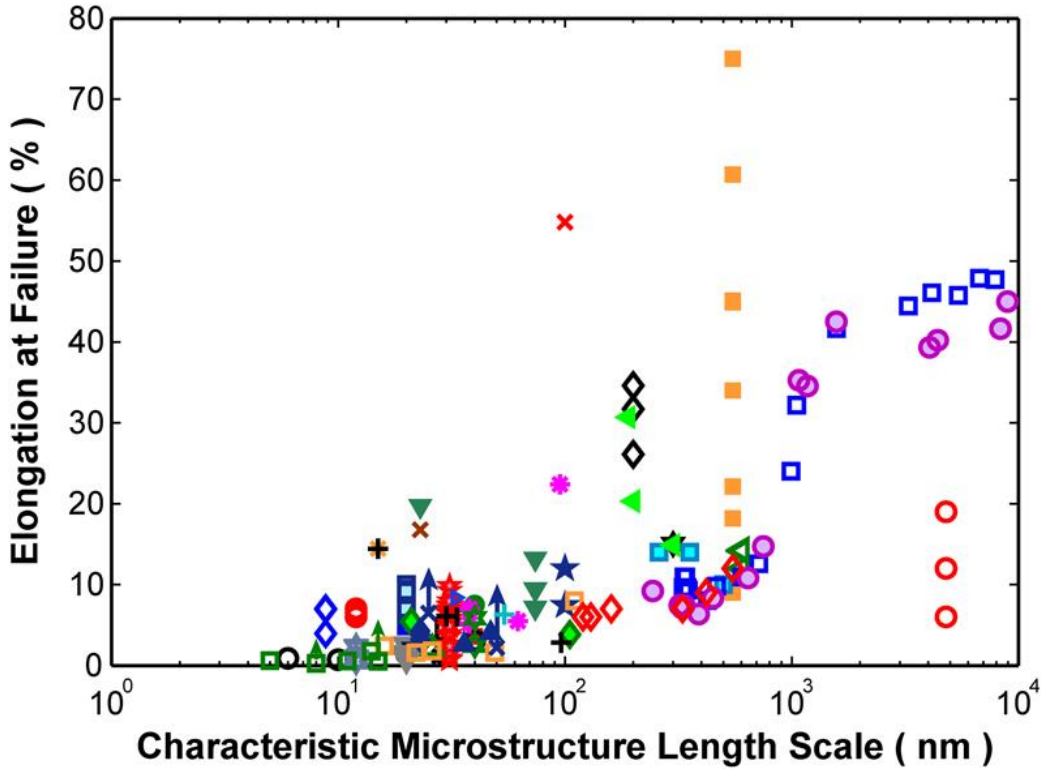

≈ 30 nm

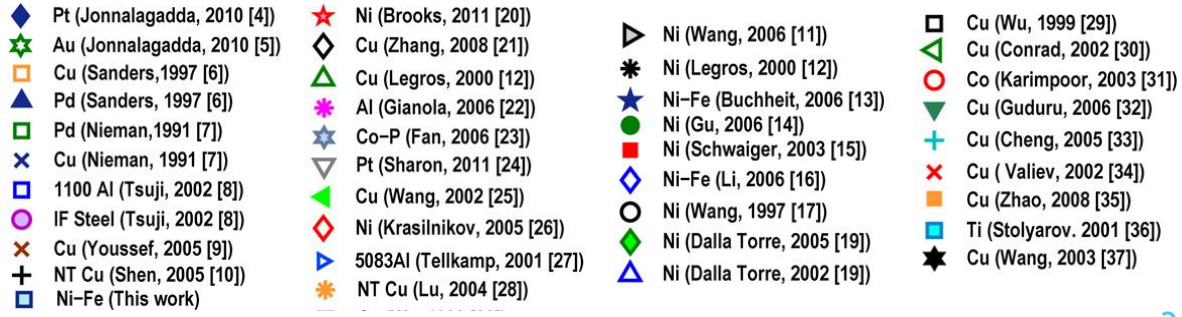



≈ 90 nm

W thin film under 650 C anneal

*Ductility*

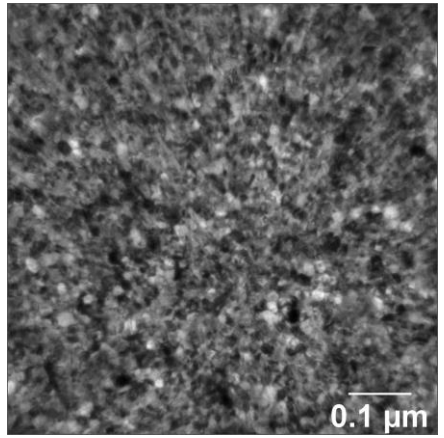


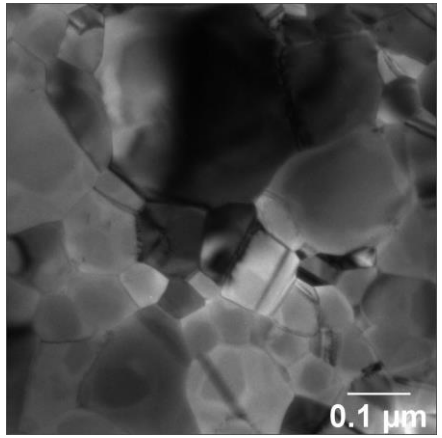




[Donaldson et al., *J. Mater. Res.*., 2017] [Sharon et al., *J. Mater. Res.*, 2013]

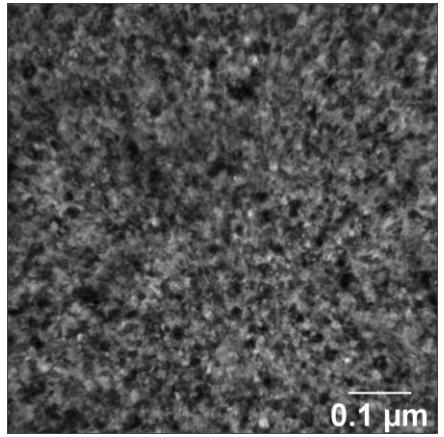
# Improved Stability with Alloying

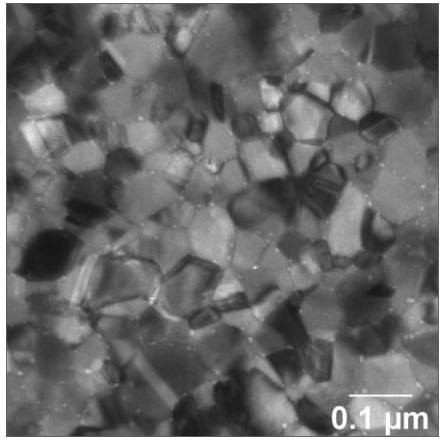
Pt




Pt-10Au



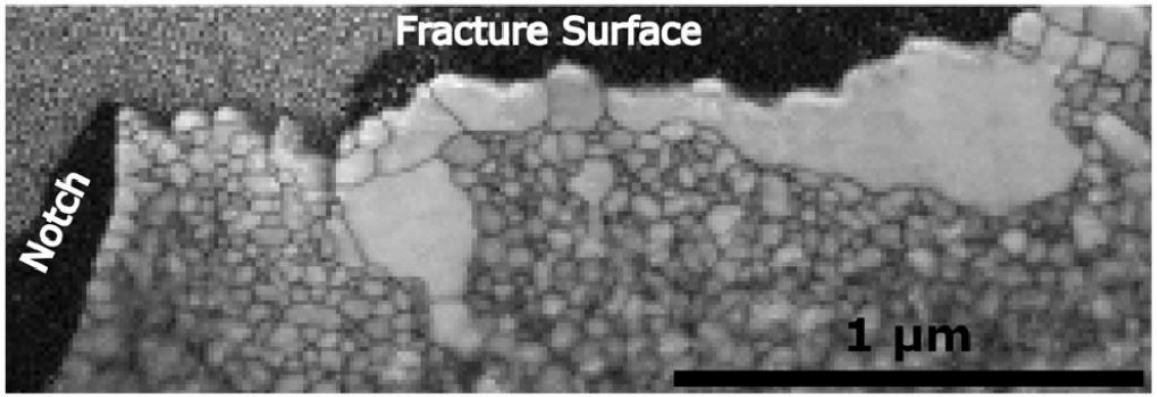




Ramp Anneal from 300 – 800 °C for 1 hour

**(c) Pt**

$N_f = 8.5 \times 10^5$ cycles

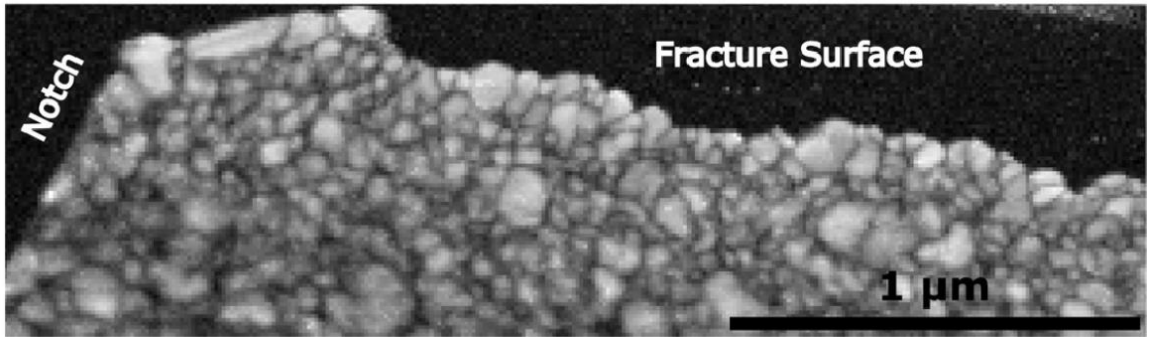
$\sigma_a = 104$ MPa




**(b) Pt-10Au**

$N_f = 7.1 \times 10^5$ cycles

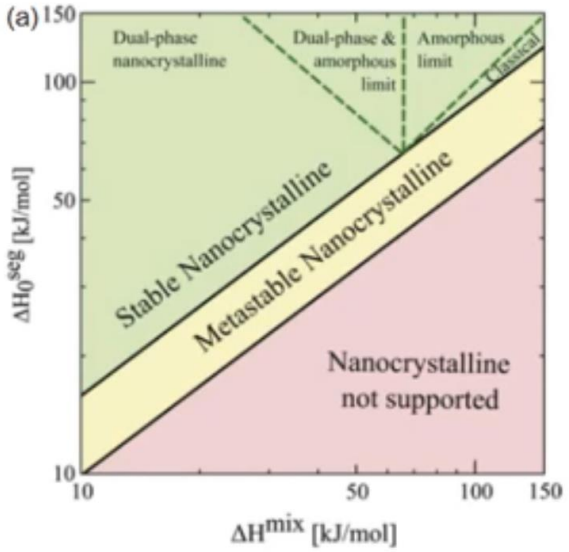
$\sigma_a = 126$ MPa




[Monti et al., *Acta Mater.*, 2022] [Heckman et al., *Acta Mater.*, 2022]

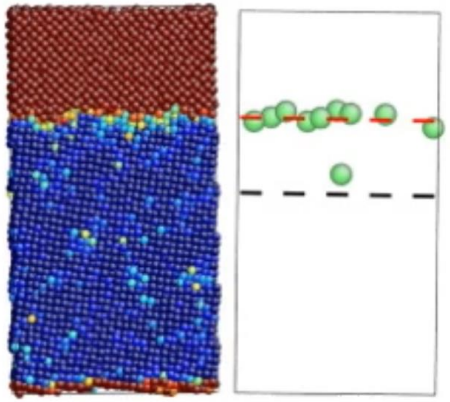
# Improved Stability with Alloying

*Thermodynamic*

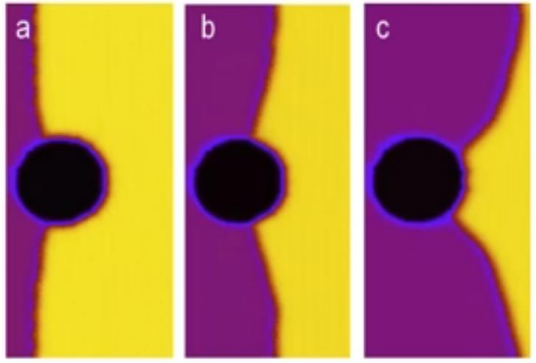



*Kinetic*

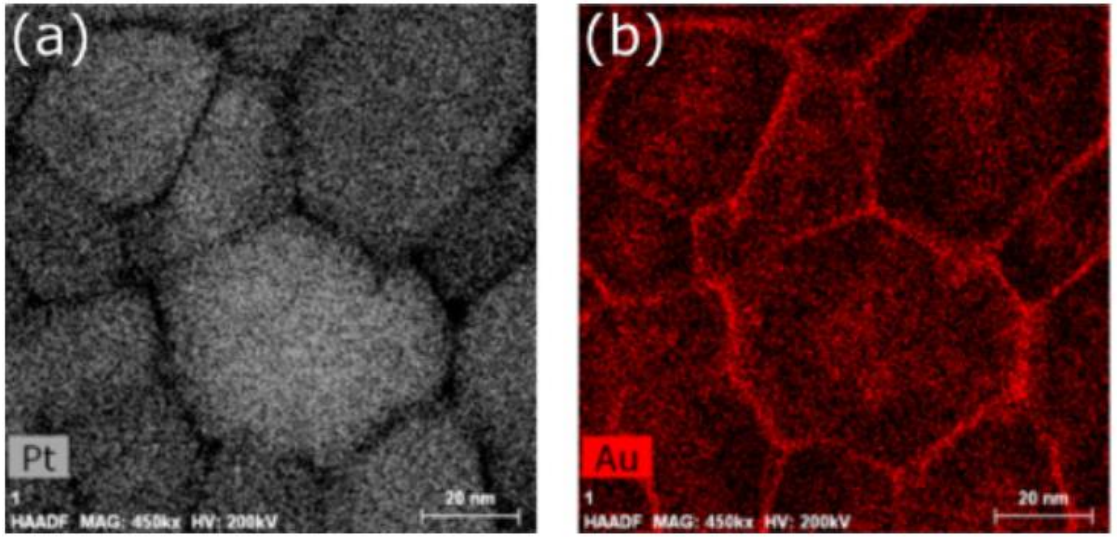
Solute drag



Zener pinning

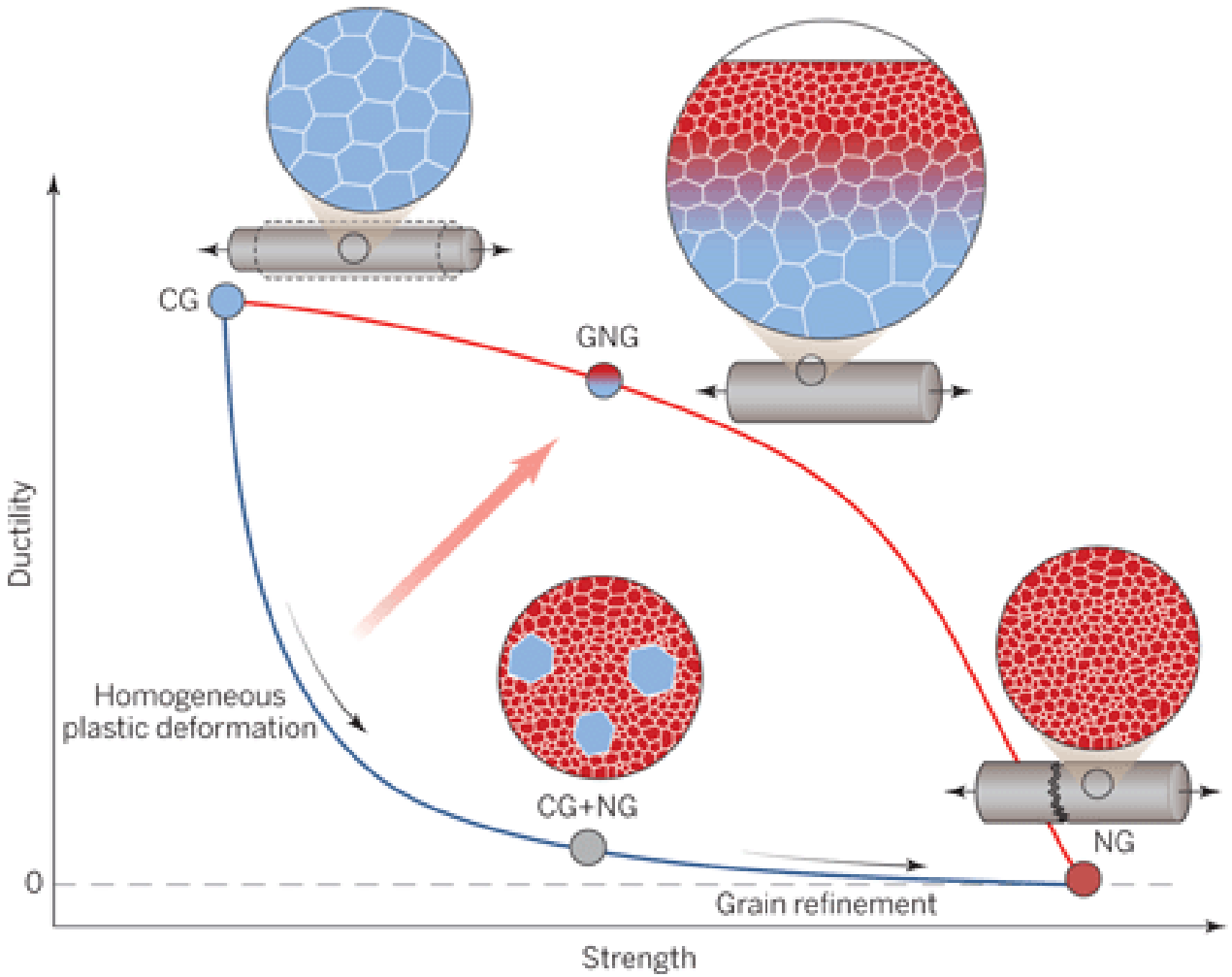


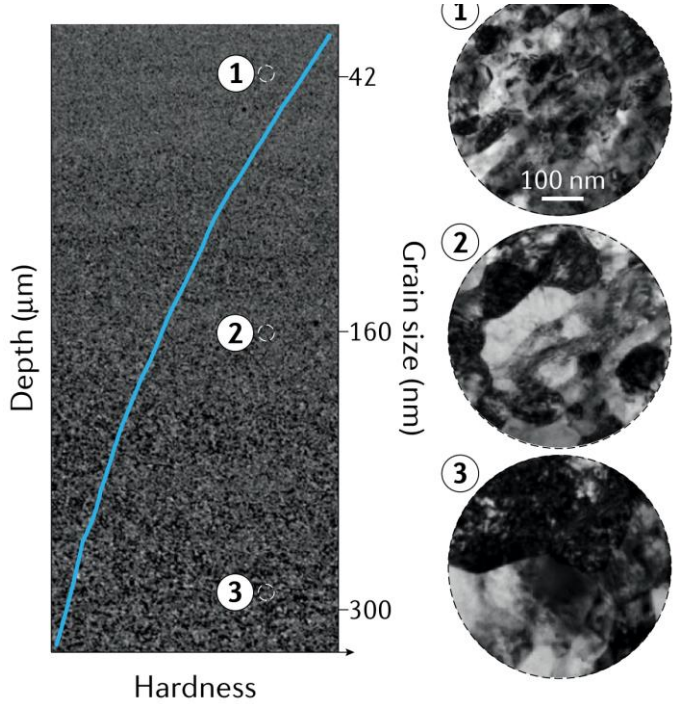

Grain Boundary Segregation



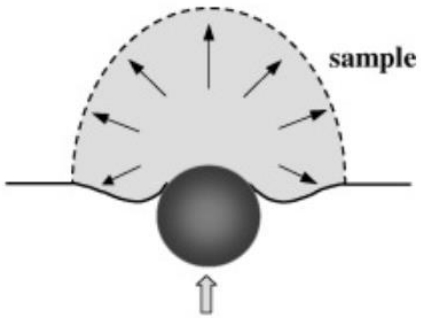

[Sun et al., *Comp. Mater. Sci.*, 2022] [Wang et al., *Acta Mater.*, 2017] [Monti et al., *Acta Mater.*, 2022]

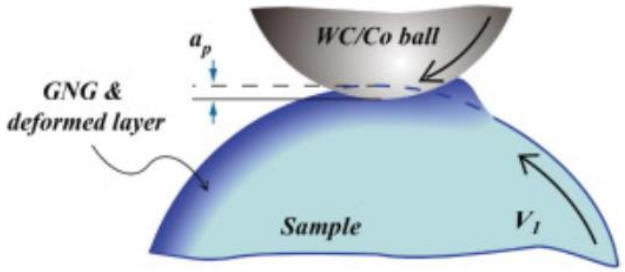
# Improved Ductility with Gradient Nanocrystalline Metals



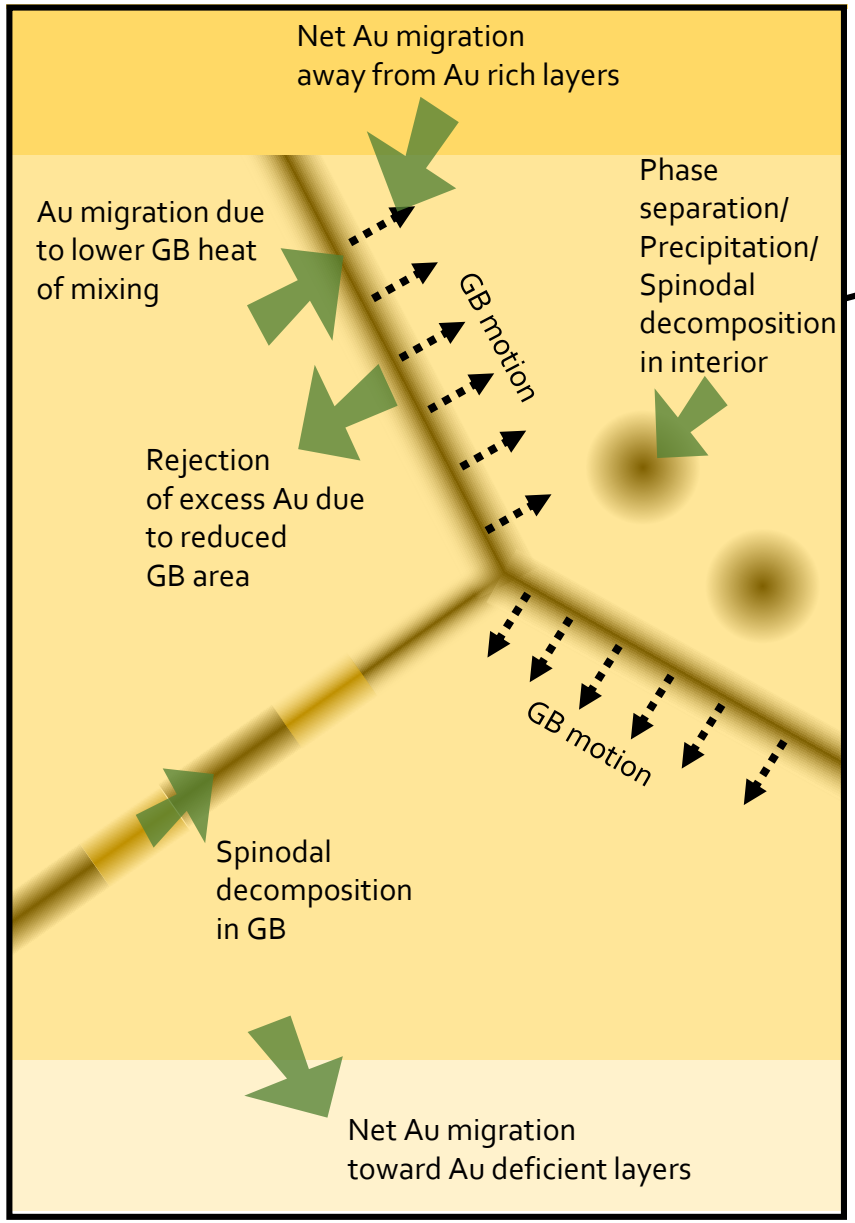

## Severe Plastic Deformation

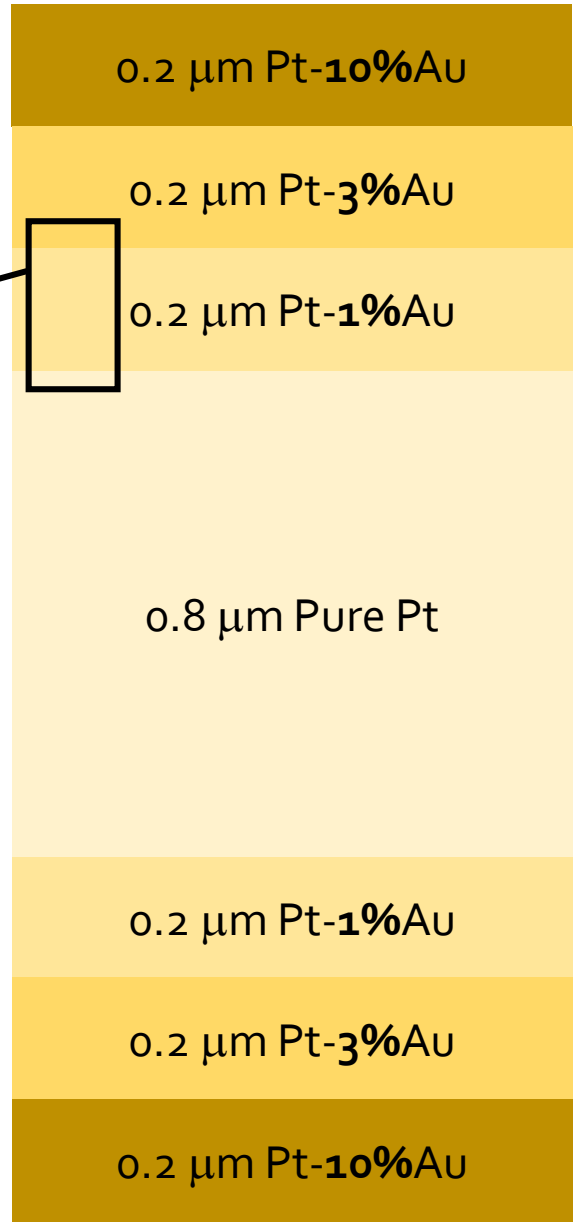


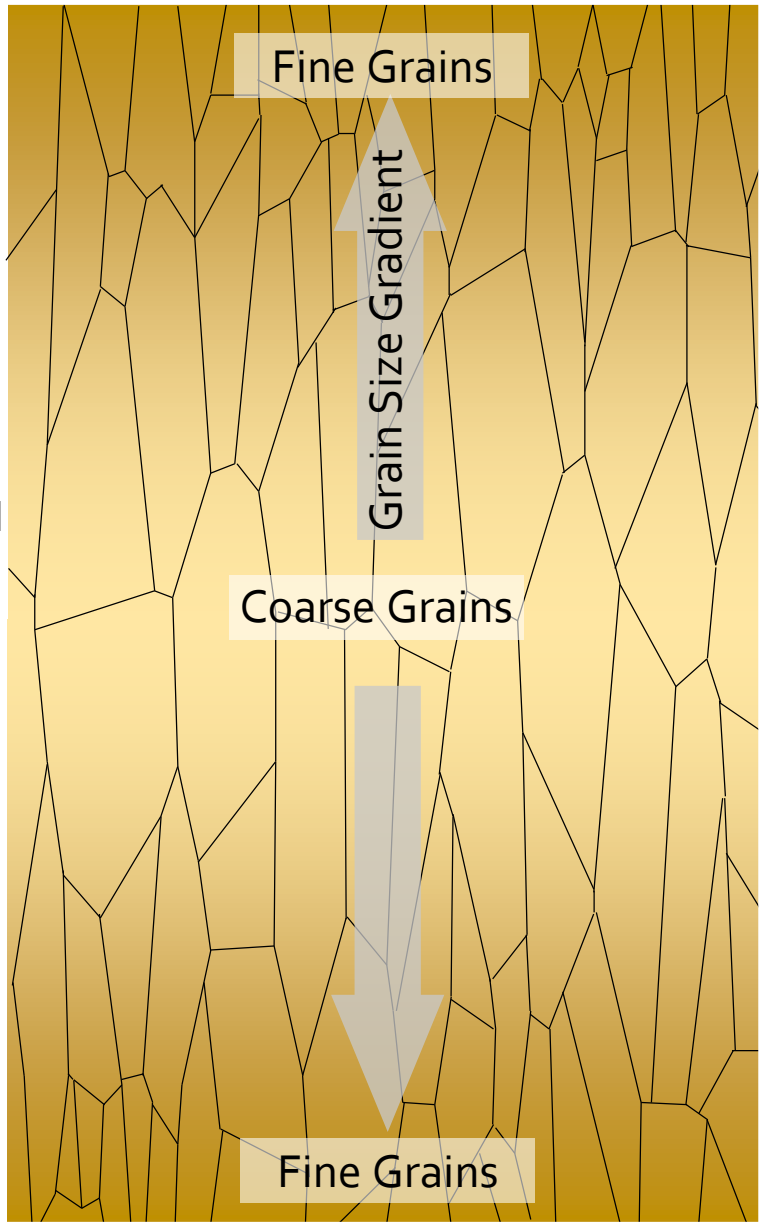

[Lu et al., *Science*, 2014] [Li et al., *Nat. Rev. Mater.*, 2020] [Lu et al., *Mater. Sci. Eng. A* ., 2004] [Huang et al., *Acta Mater.*, 2015]

# Gradient Nanostructured Metals via Compositional Means



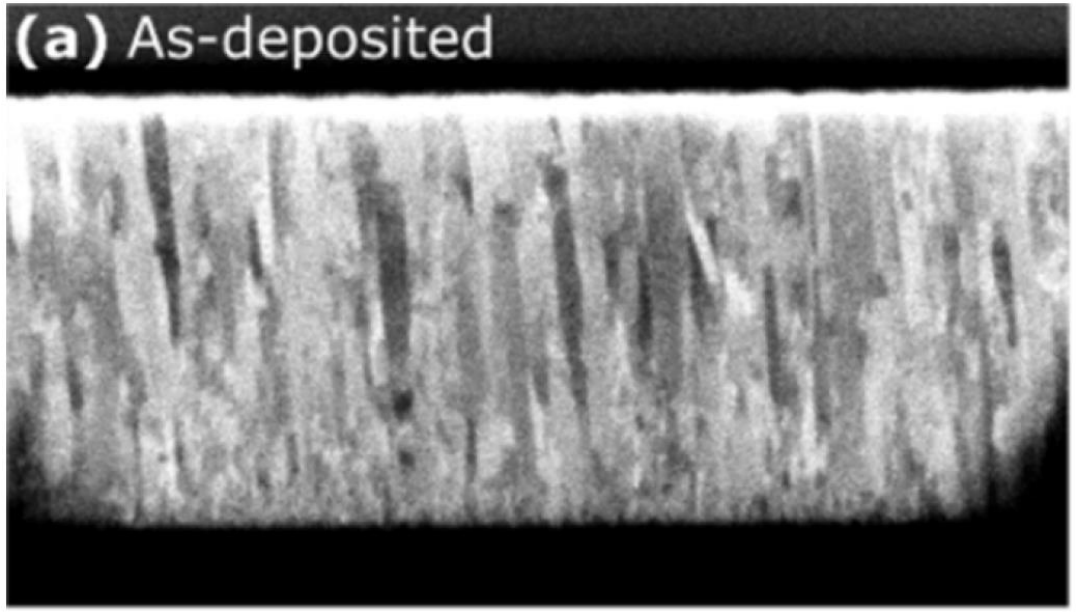




Anneal




[Barrios et al., *Acta Mater.*, 2023]

# As-deposited sample




7 layer stack by sequential DC magnetron sputeering

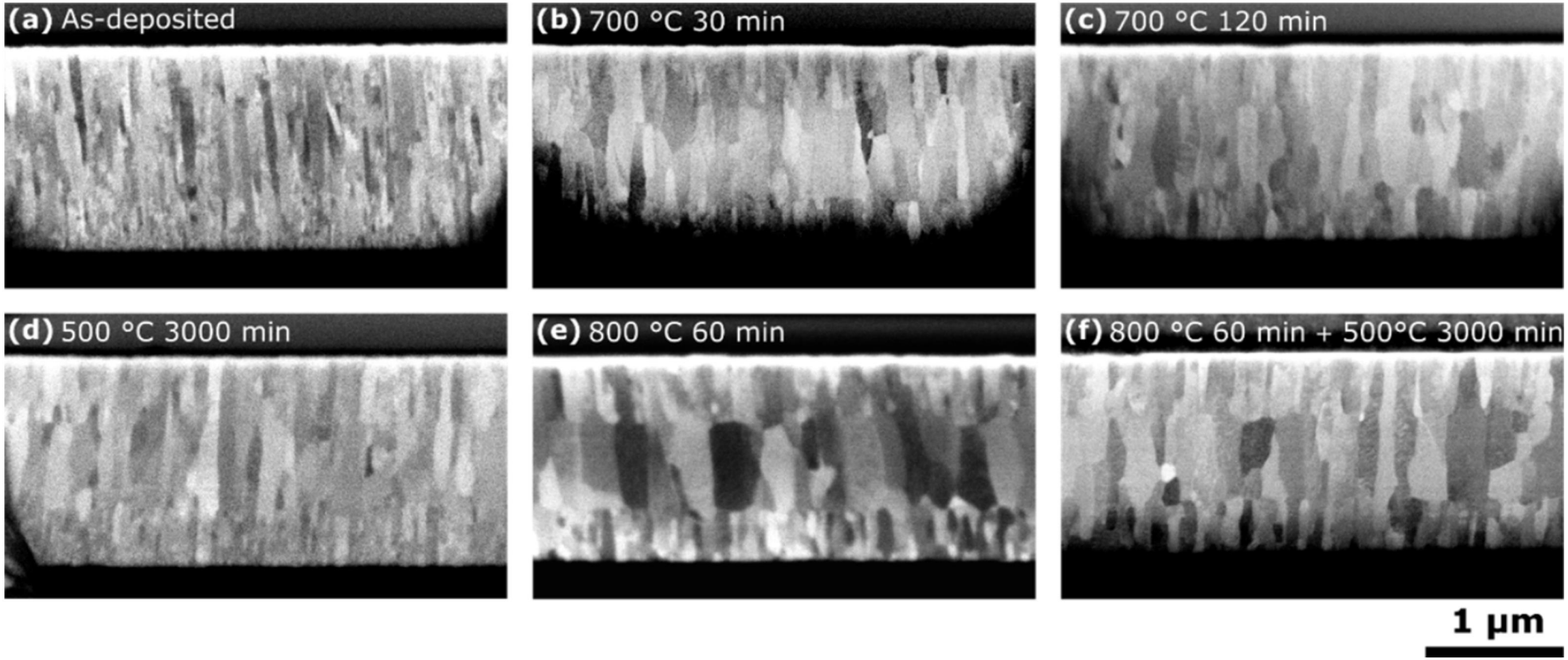
≈2 um thick film

Annealing experiments in a box furnace
- Temperature varying from 500 – 1000 C
- Times varying from 5 minutes to 50 hours

FIB cross-section and backscatter contrast images to study microstructure in the cross-section of the films

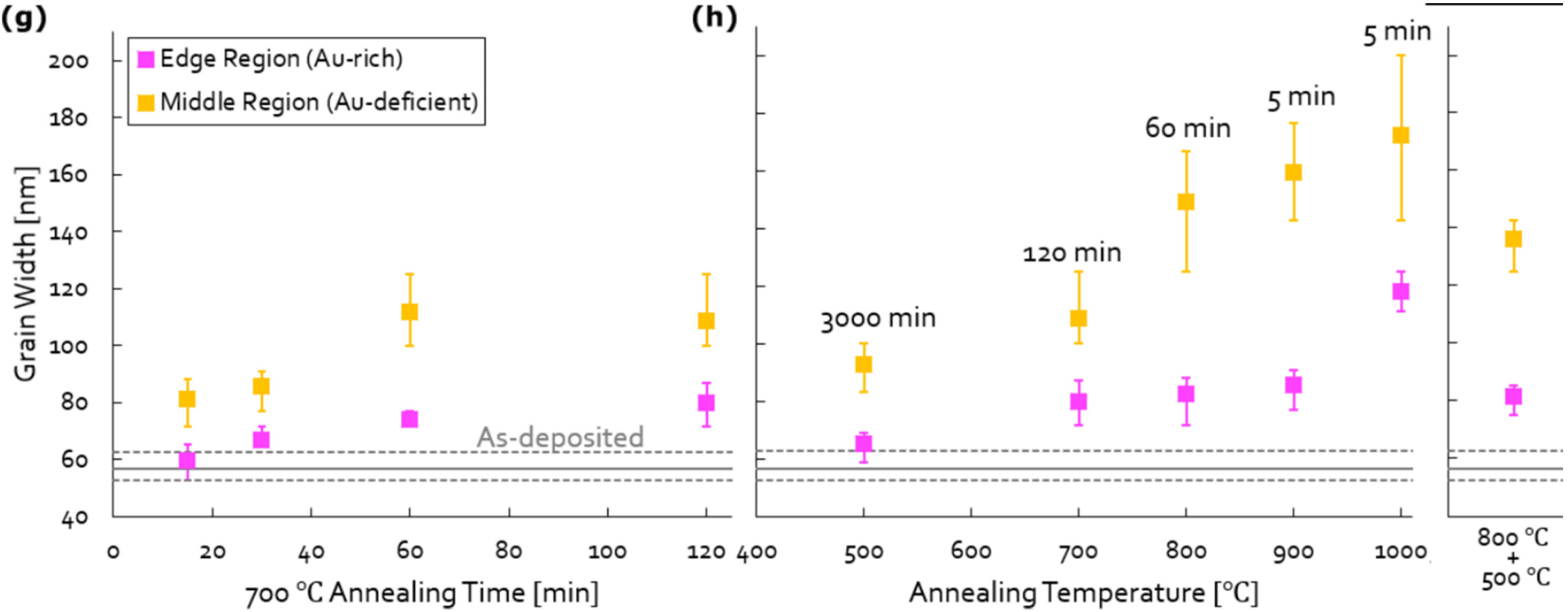
[Barrios et al., *Acta Mater.*, 2023]

# Annealing Experiments



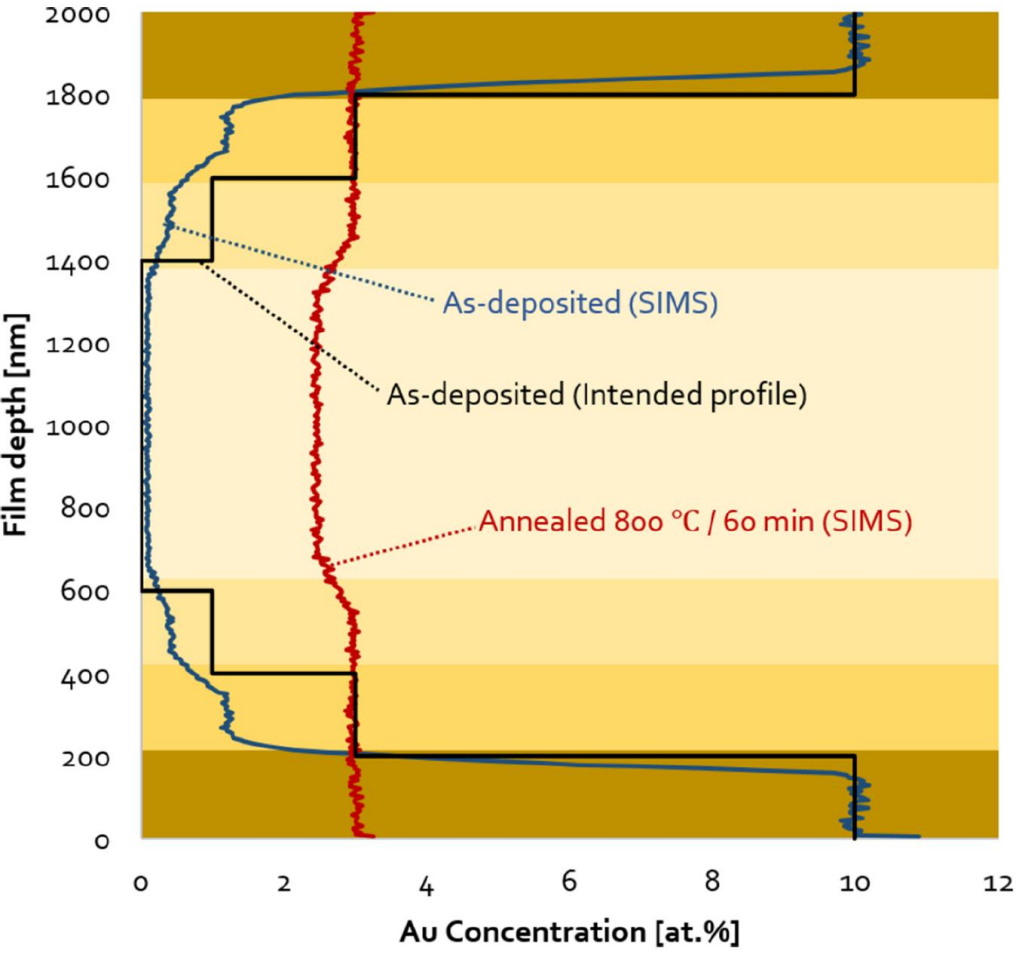

[Barrios et al., *Acta Mater.*, 2023]

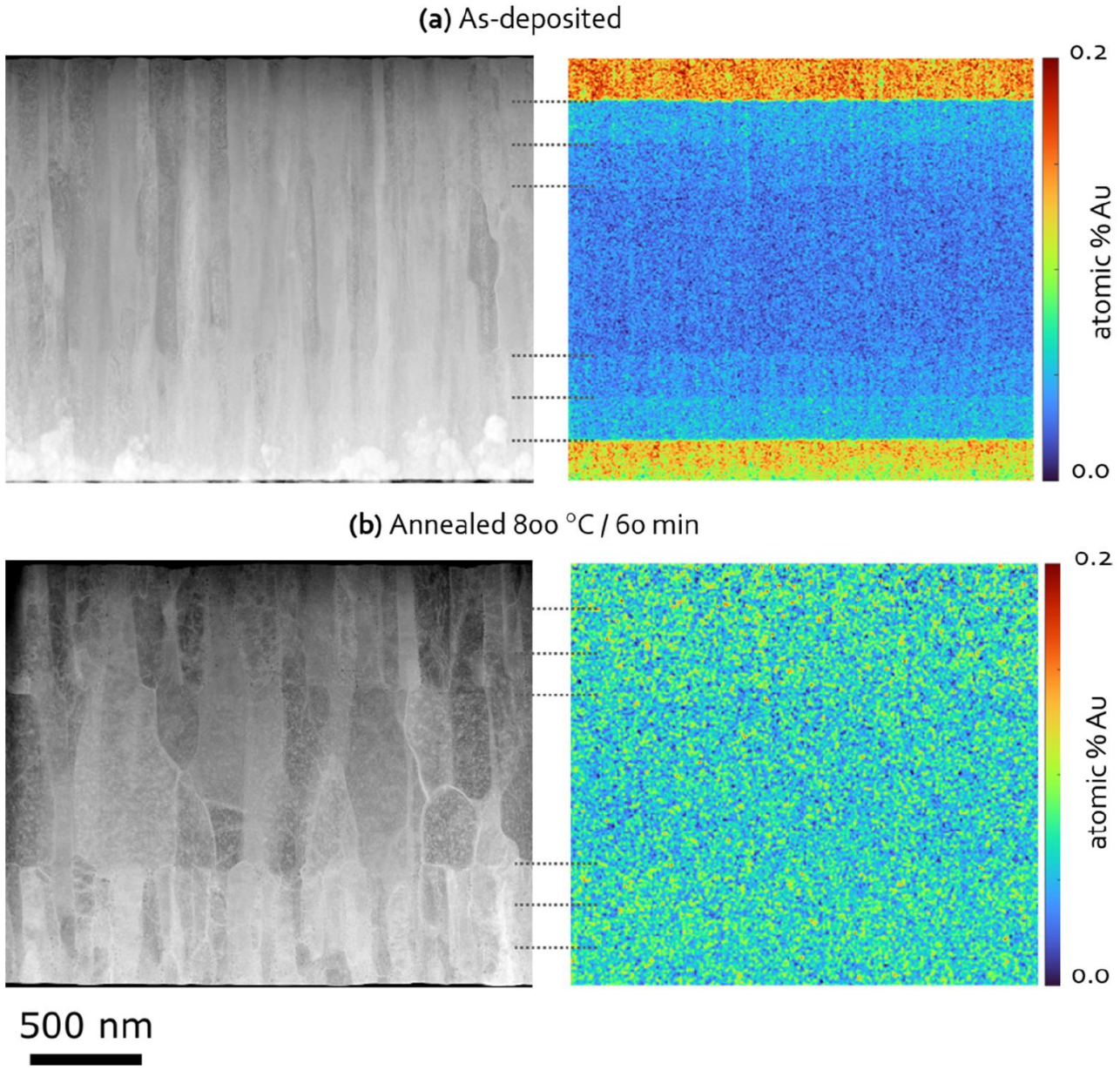
# Annealing Experiments




[Barrios et al., *Acta Mater.*, 2023]

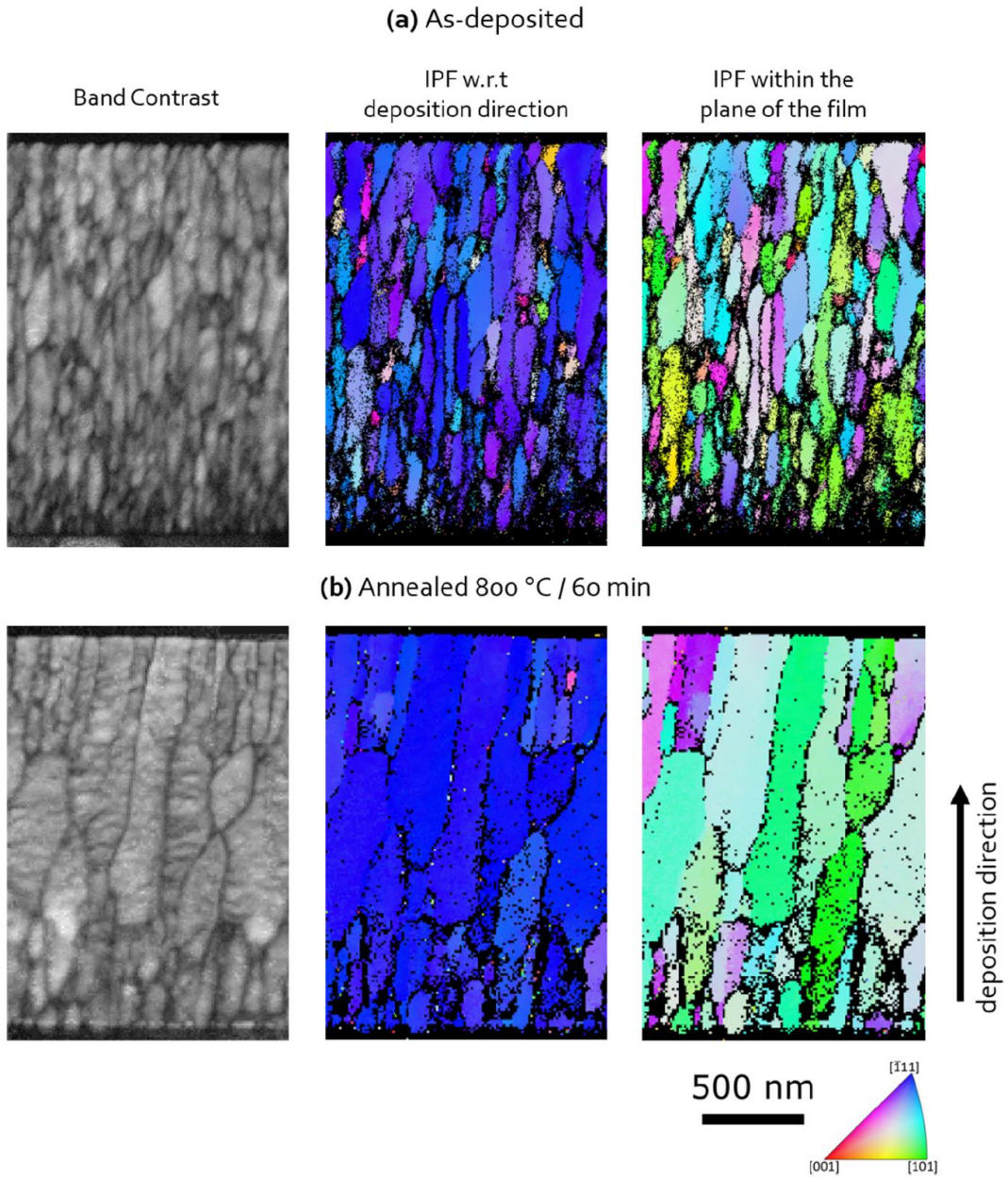
# Film Characterization – SIMS/STEM/EDS

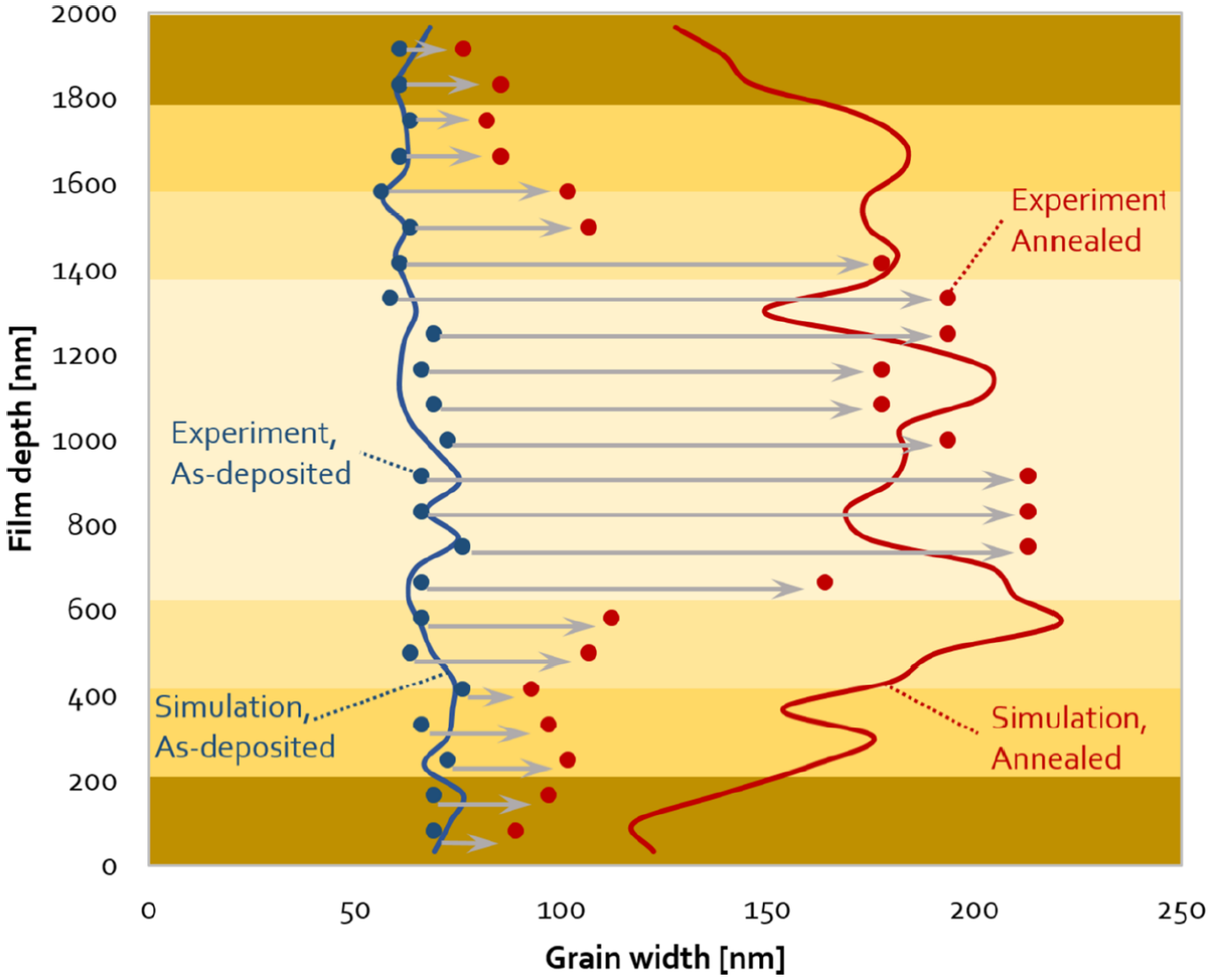



[Barrios et al., *Acta Mater.*, 2023]

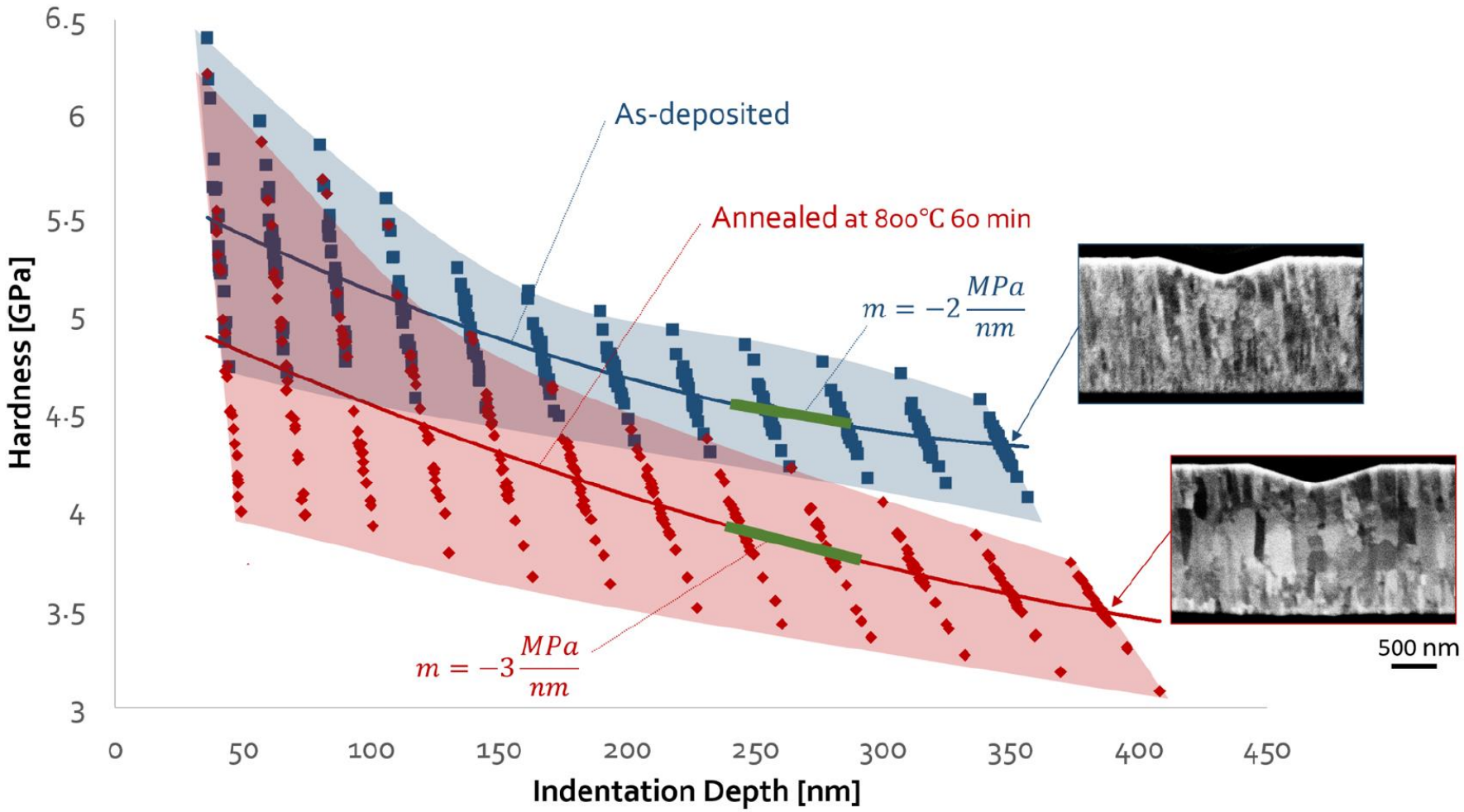
# Film Characterization - TKD




[Barrios et al., *Acta Mater.*, 2023]

# Film Characterization - Nanoindentation

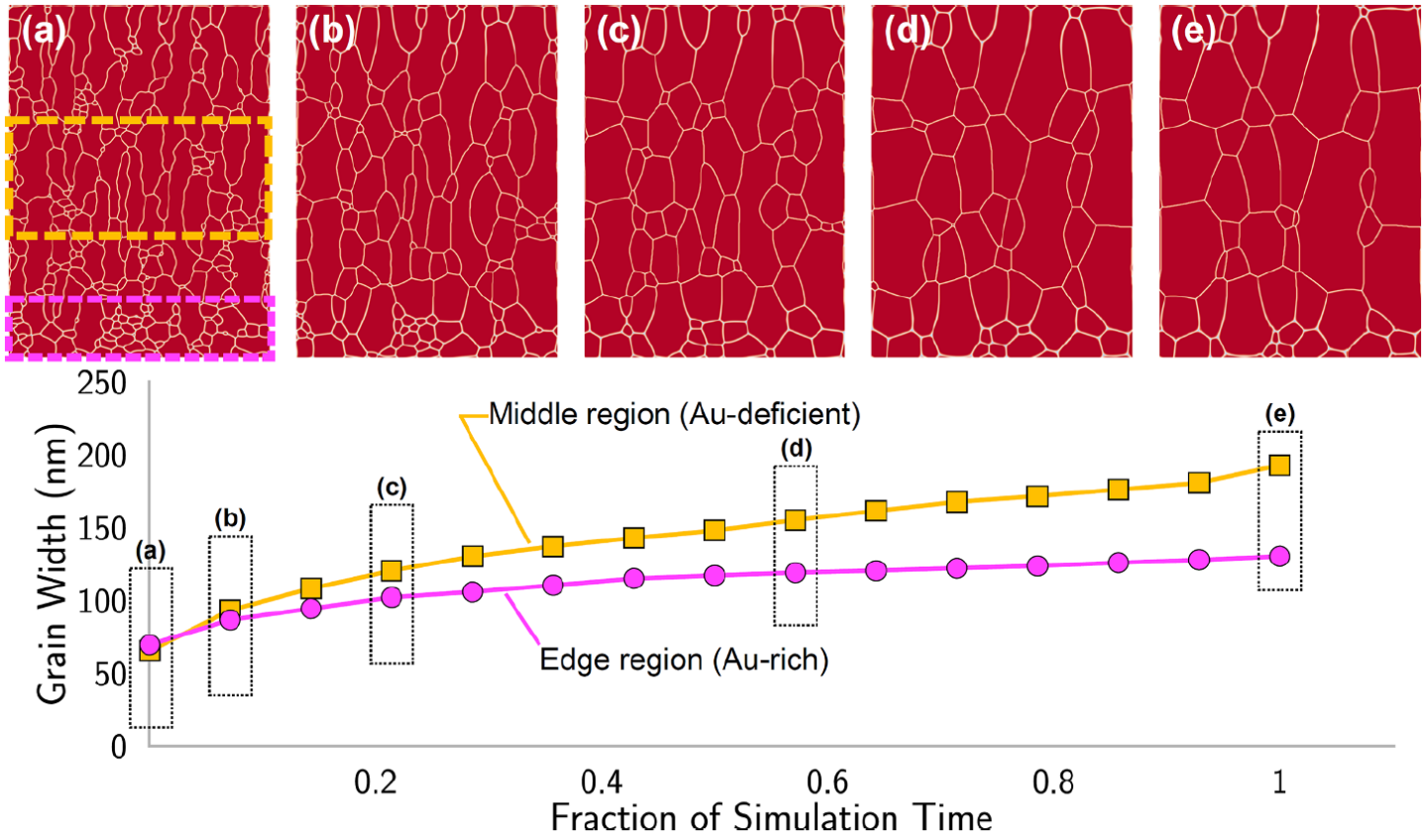


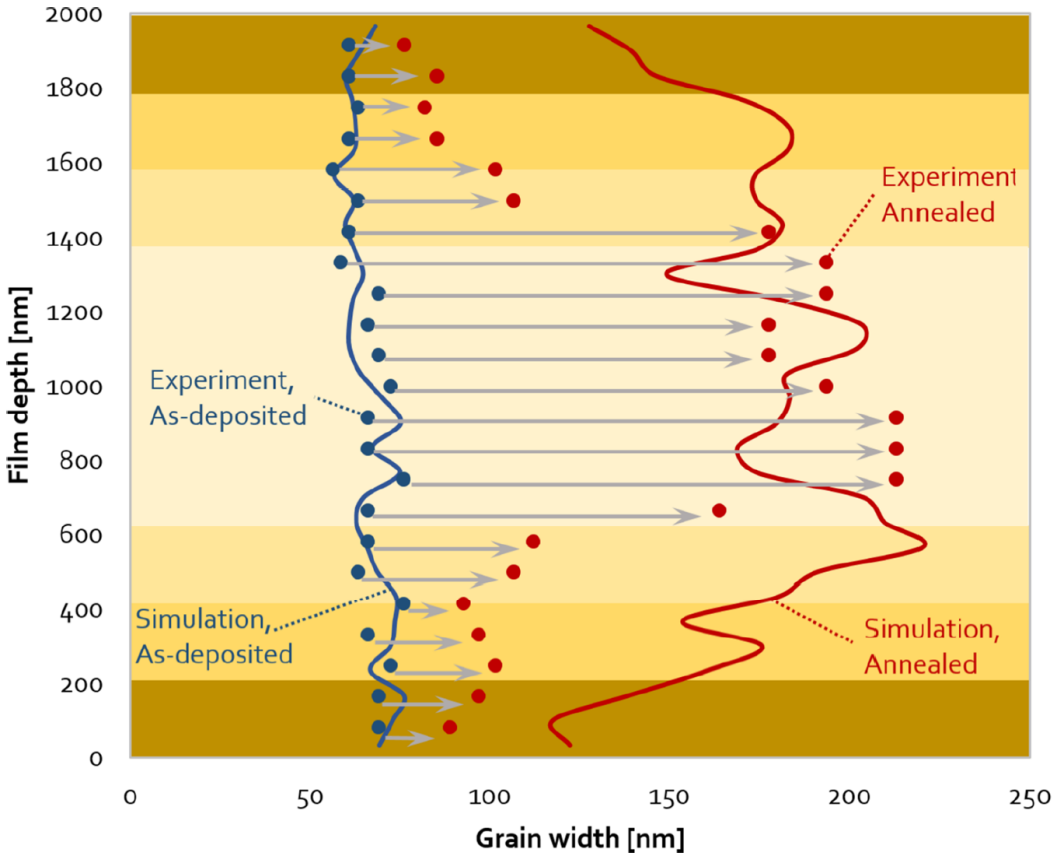

Gradient hardness trend for as-deposited case is a consequence of the gradient solute content

Steeper hardness trend for annealed case is a consequence of the gradient grain size

[Barrios et al., *Acta Mater.*, 2023]

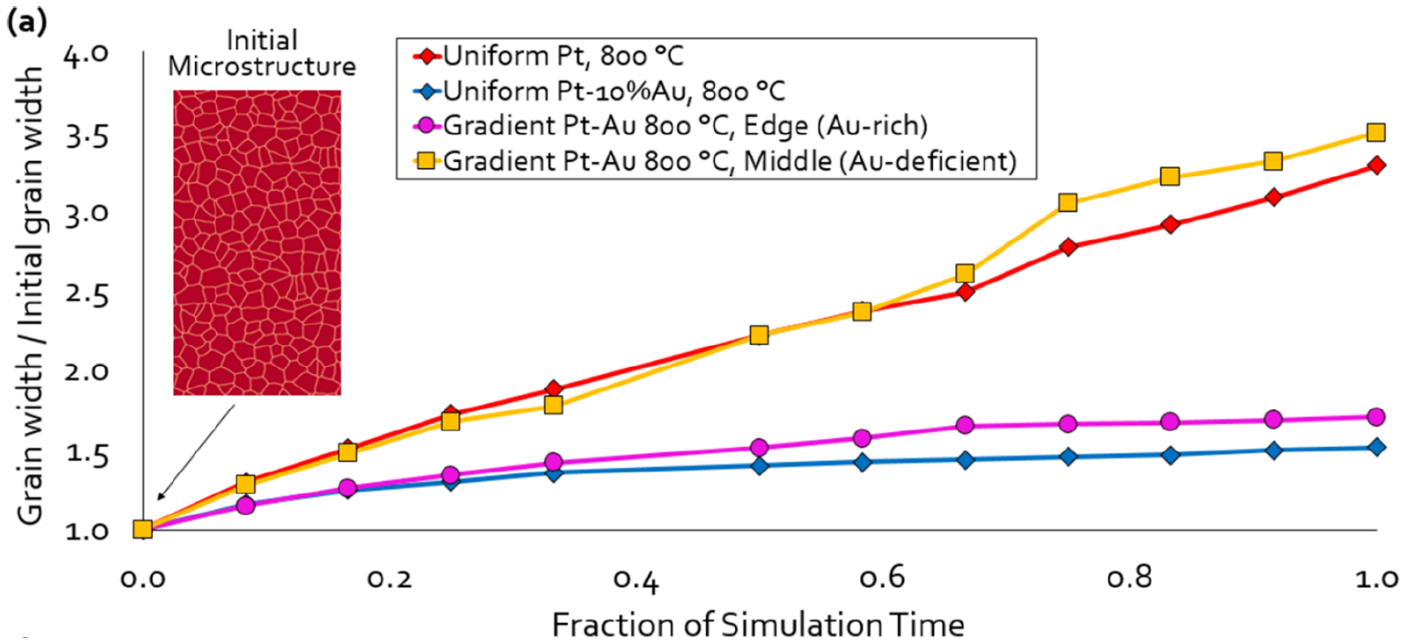
# Phase-field Modeling

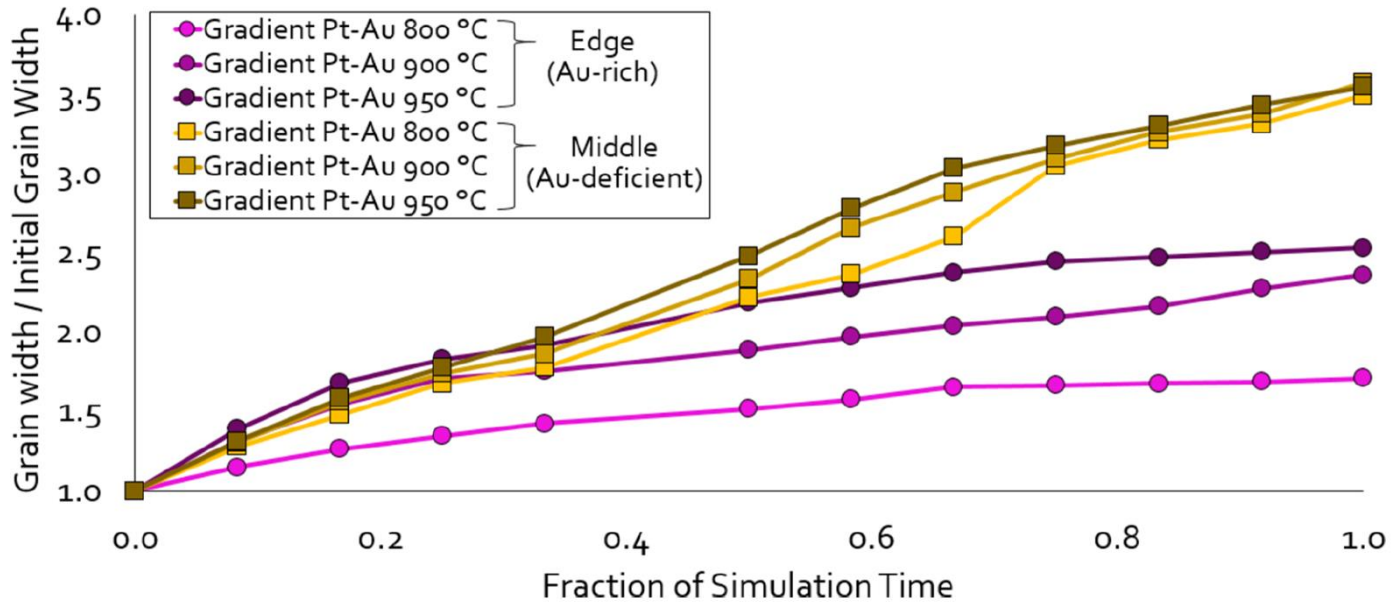



[Barrios et al., *Acta Mater.*, 2023]

# Phase-field Modeling – Expansion to Equiaxed Microstructure and Different Temperatures

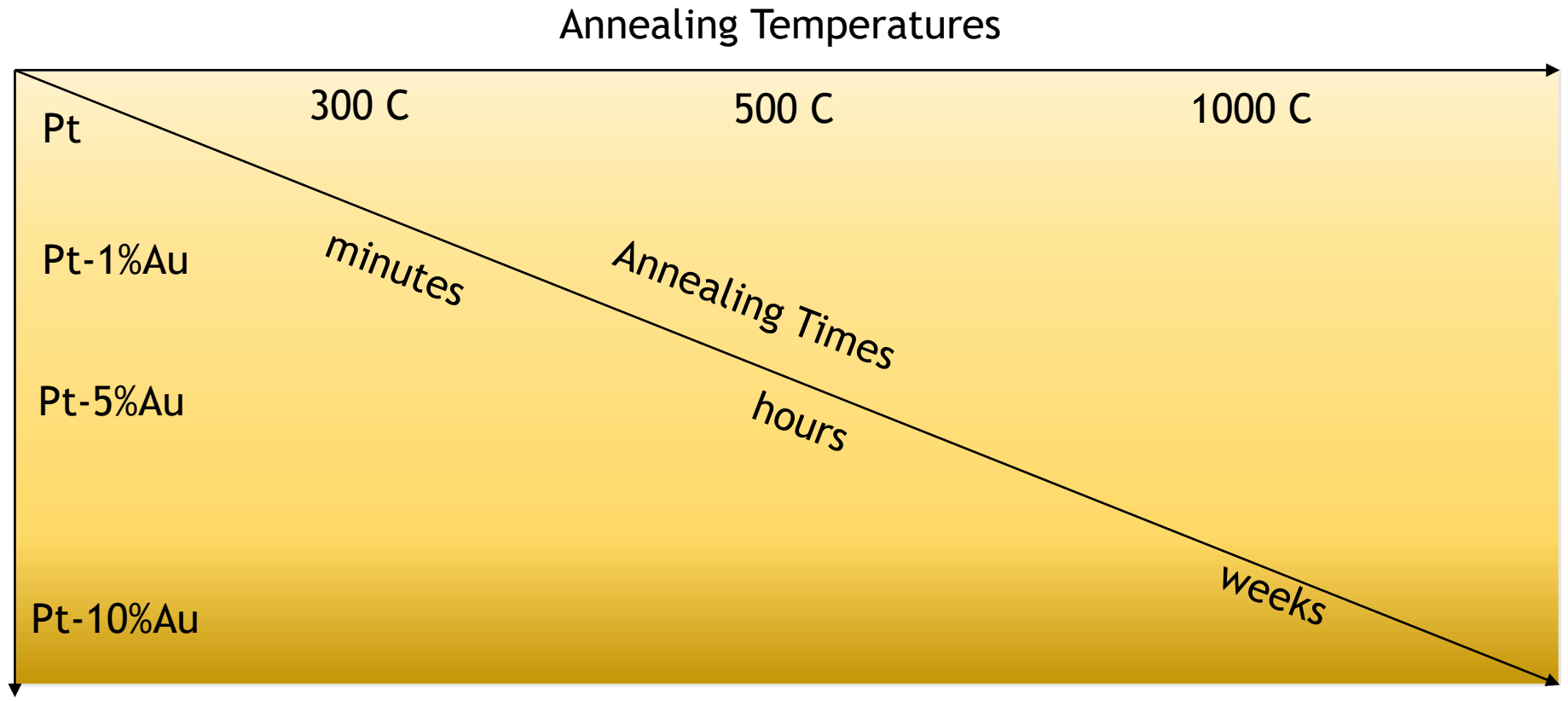


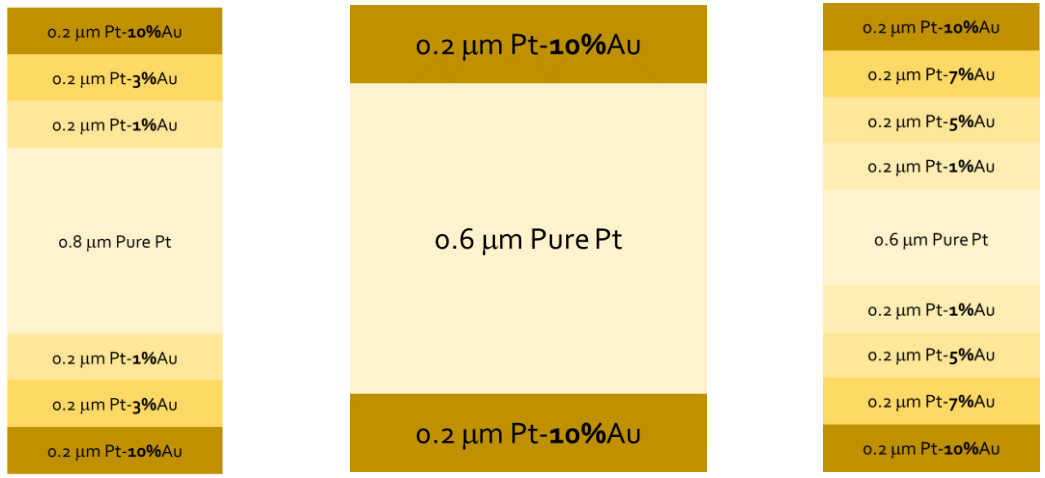

[Barrios et al., *Acta Mater.*, 2023]

# Design Space Exploration with Phase-Field Modeling

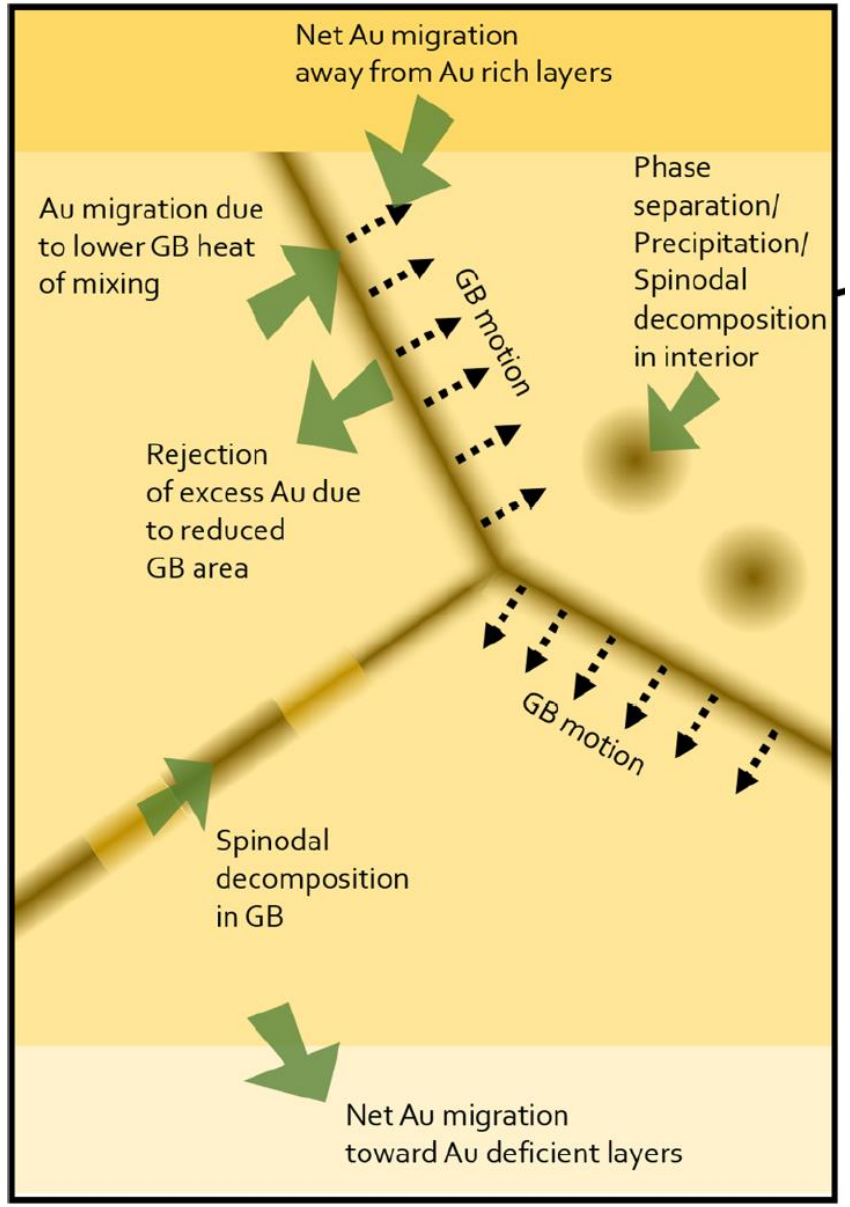
Homogeneous compositions




Layered compositions



# Mechanisms




Six kinetic processes contributing to the simultaneous evolution of the grain structure and the spatial composition gradients.

Segregation of Au to grain boundaries is able to impede grain growth

Au starts diffusing into the middle of the film, further grain growth in that region becomes suppressed.

Before the Au arrives, the grains in the middle of the film are allowed to coarsen under high temperature

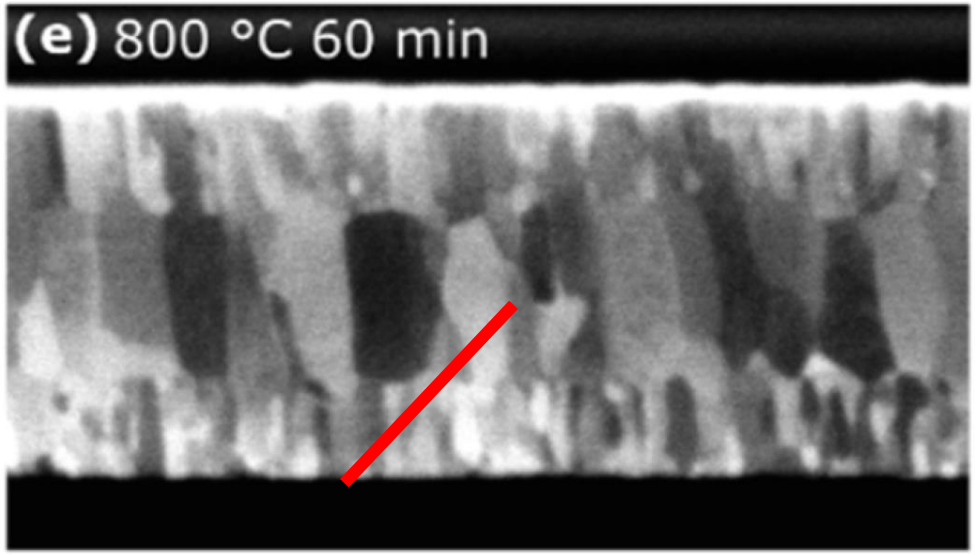
At the edges, with high Au concentration, the grains coarsen at much slower rates

The overarching result is a stable gradient nanostructured film with a more uniform composition.

[Barrios et al., *Acta Mater.*, 2023]

# Advantages of Gradient Nanostructured Metals via Compositional Means

Grain size gradient in other studies: ≈1-10 nm/um

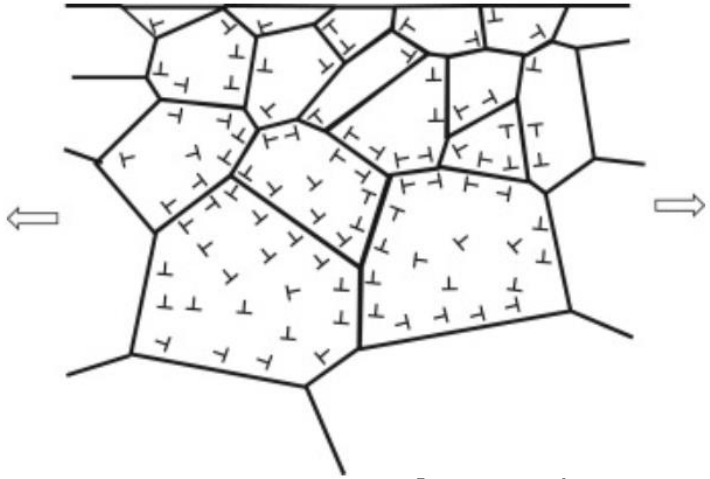



Grain size gradient: ≈100 nm/um

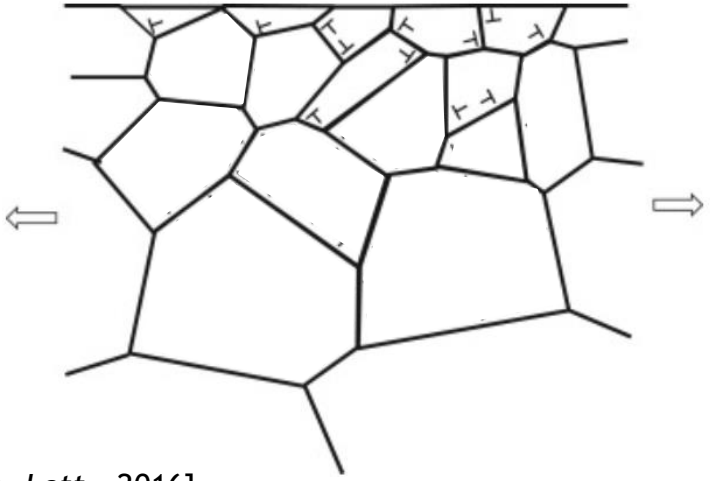
Controlled by varying annealing times and temperatures

Confine strain localization in a smaller volume

Gradient structure via severe plastic deformation



Gradient structure via compositional means

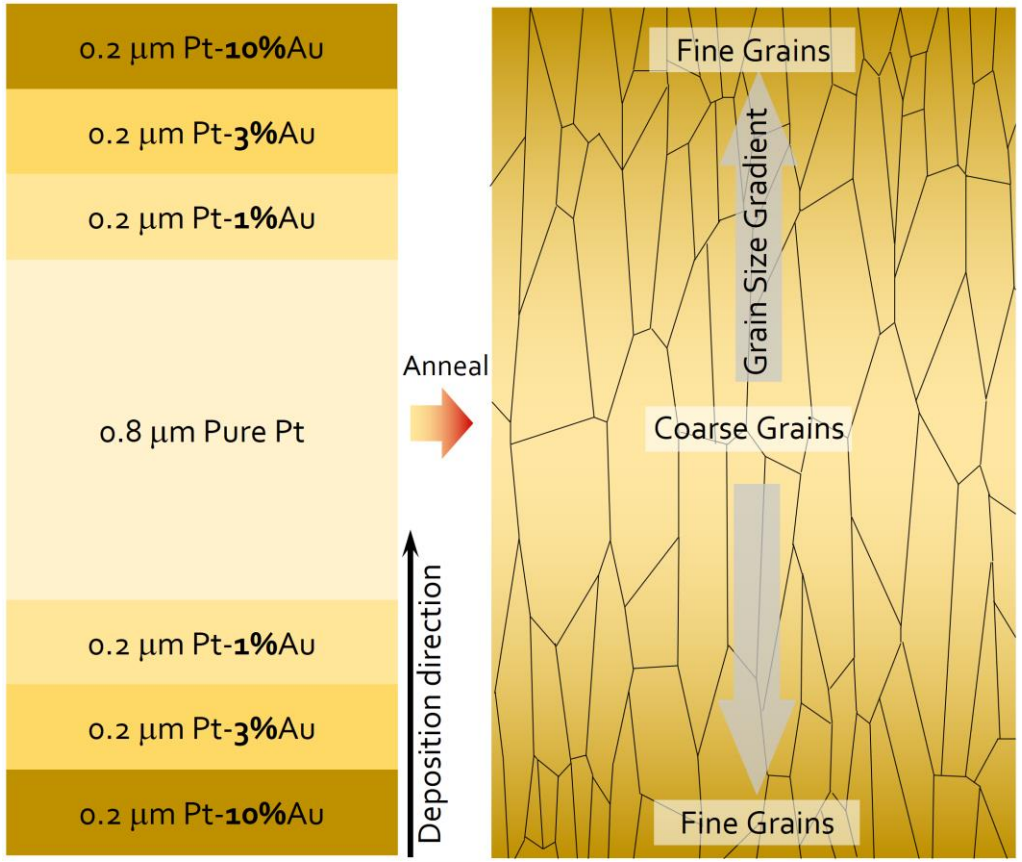


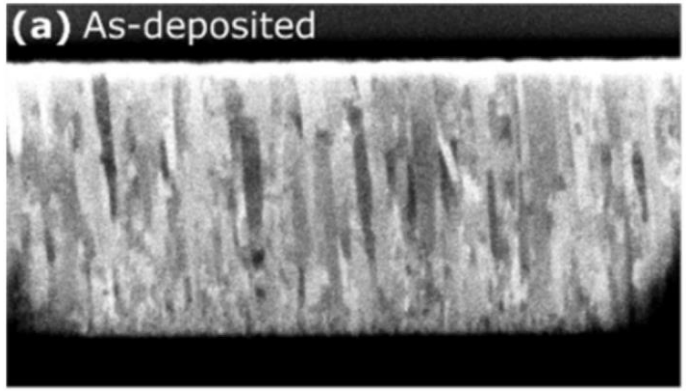
Fully recrystallized microstructure
- Higher degree of possible strain hardening

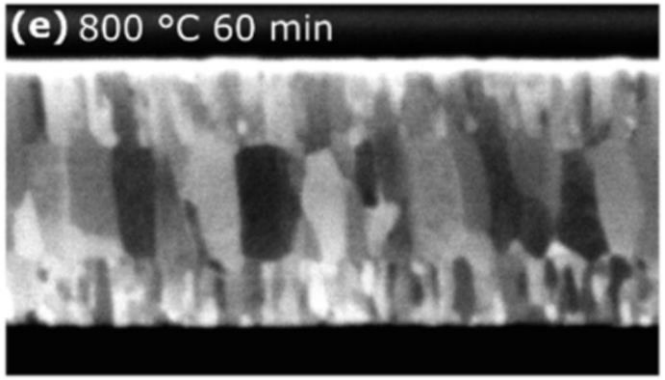
Future Work:
- Tensile (explore strengthening)
- Fatigue (explore grain growth and stability)

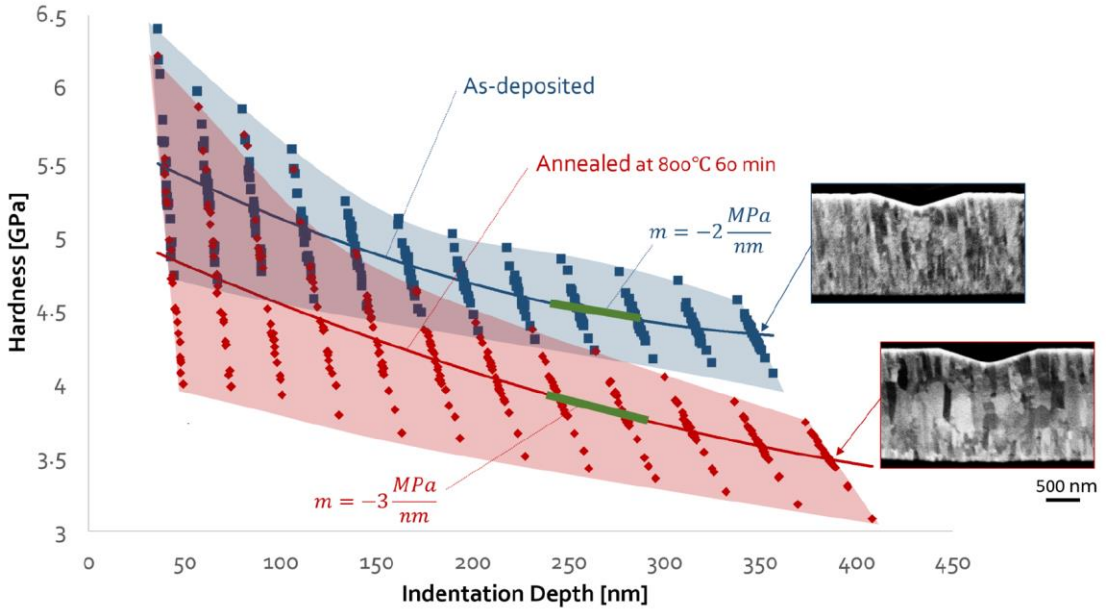
[Barrios et al., *Acta Mater.*, 2023] [Zeng et al., *Extreme Mech. Lett.*, 2016]

# Conclusions



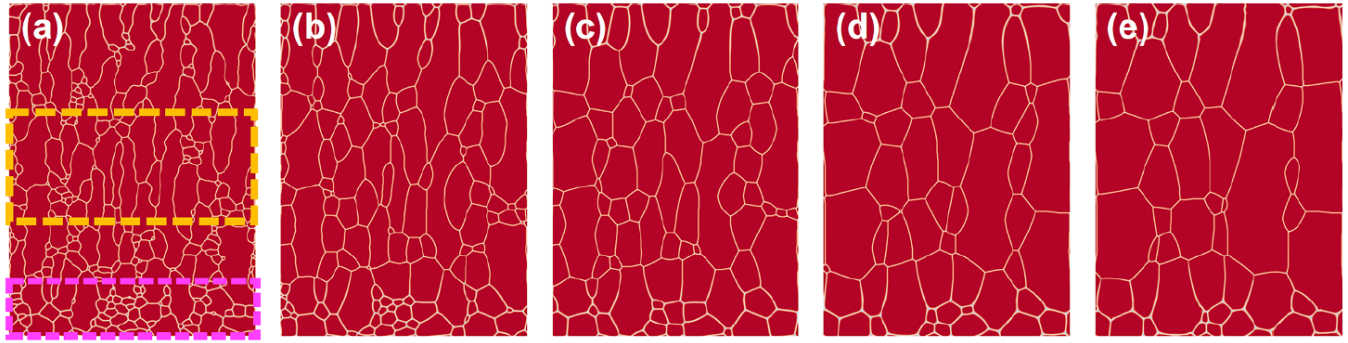



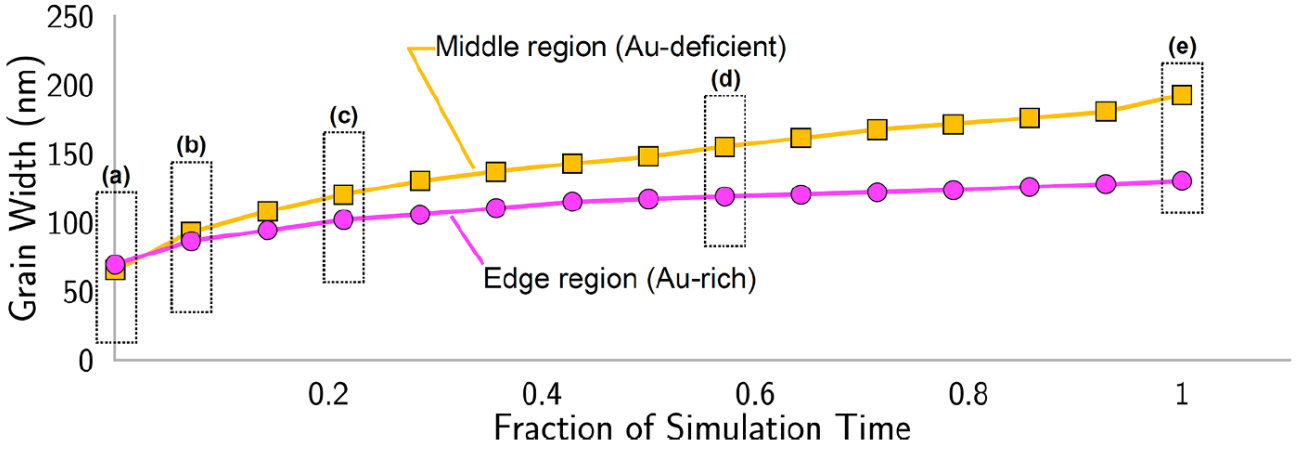




[Barrios et al., *Acta Mater.*, 2023]

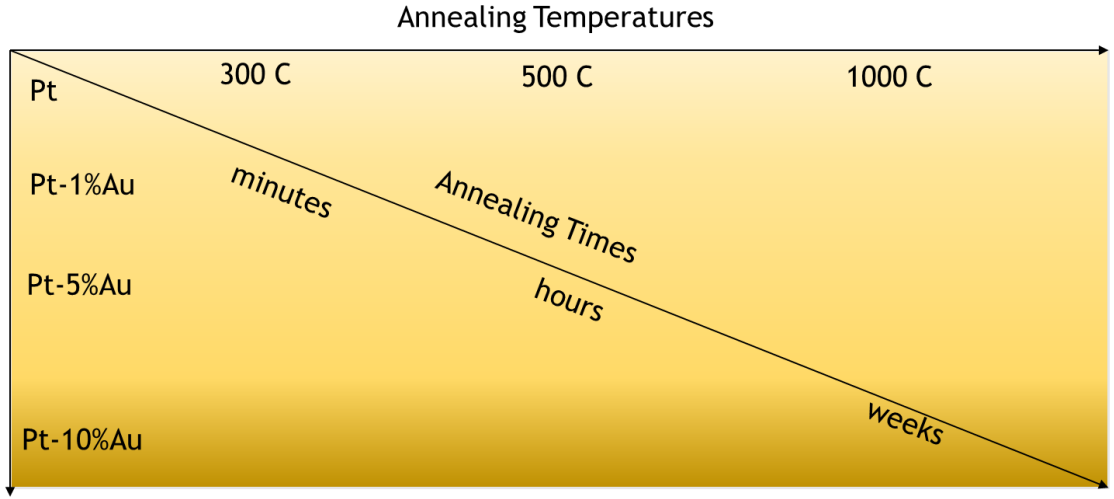
# Conclusions

## Phase-field modeling to explore design space




Homogeneous compositions




[Barrios et al., *Acta Mater.*, 2023]