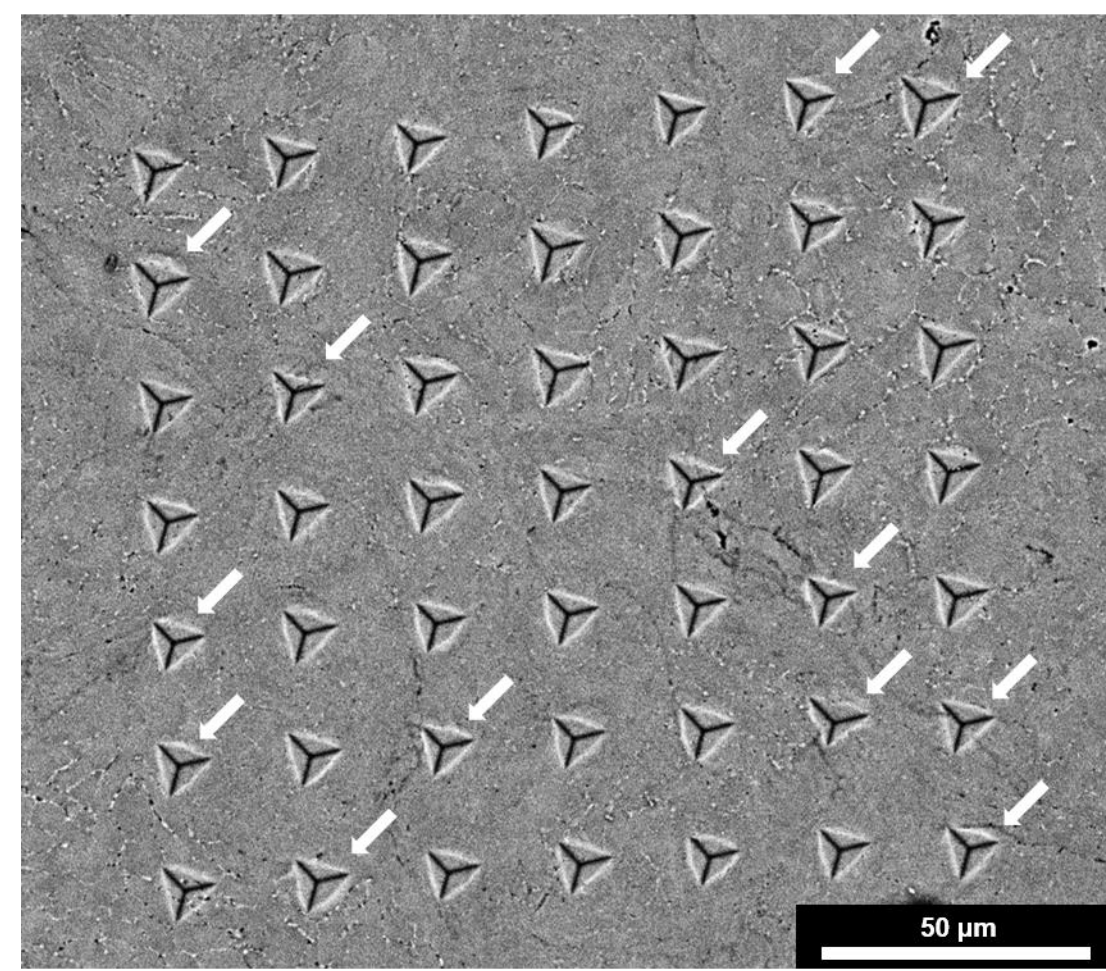


## Introduction

- The cold spray process involves the acceleration of powder material to supersonic velocities using a high pressure gas jet.
- The process typically produces a unique duplex microstructure in which the centers of the constituent particles are comprised of an equiaxed grain structure, while the particle exterior undergoes significant deformation and recrystallization leading to a fine, squashed grain structure.
- It is important to investigate the mechanical properties and microstructural evolution due to heat-treatment of these distinct regions to understand how they influence the mechanical properties of the resultant cold-sprayed coatings.**

## Al-6061 Indentation

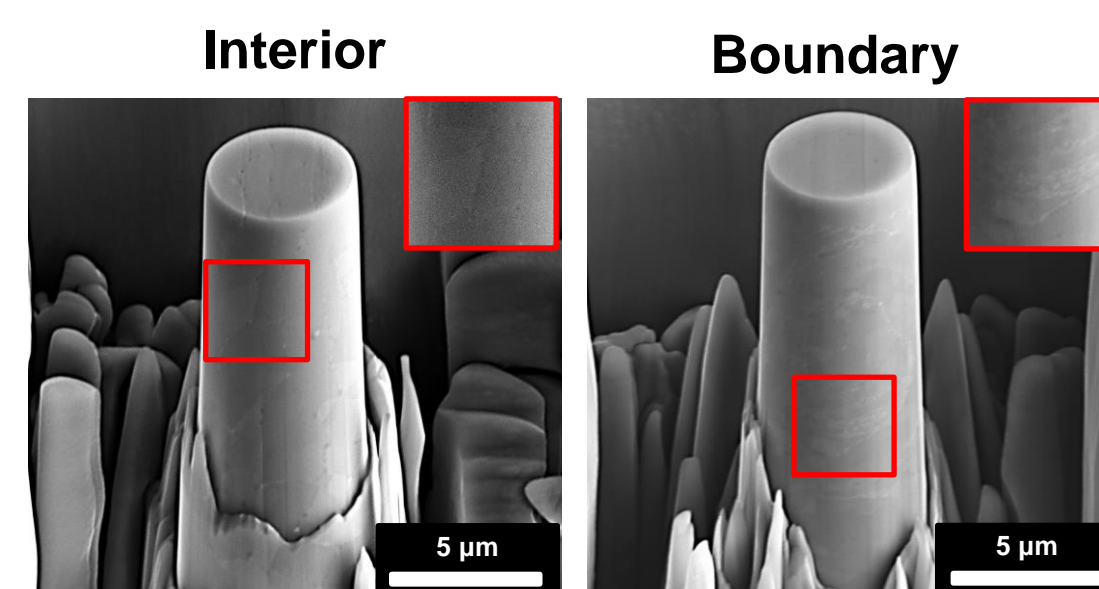
- Samples were cut from discarded tensile bars of Al6061 produced by cold spray perpendicular and parallel to the spray direction.
- A series of isothermal heat-treatments ranging from 100-500°C for 2 hours were performed.
- Nano-indentation was utilized to examine the microhardness of the interior and boundary regions.
- Vickers hardness testing was also performed for comparison.



Array of indents on as-received deposit parallel to the spray direction (whit arrows indicate boundary region)

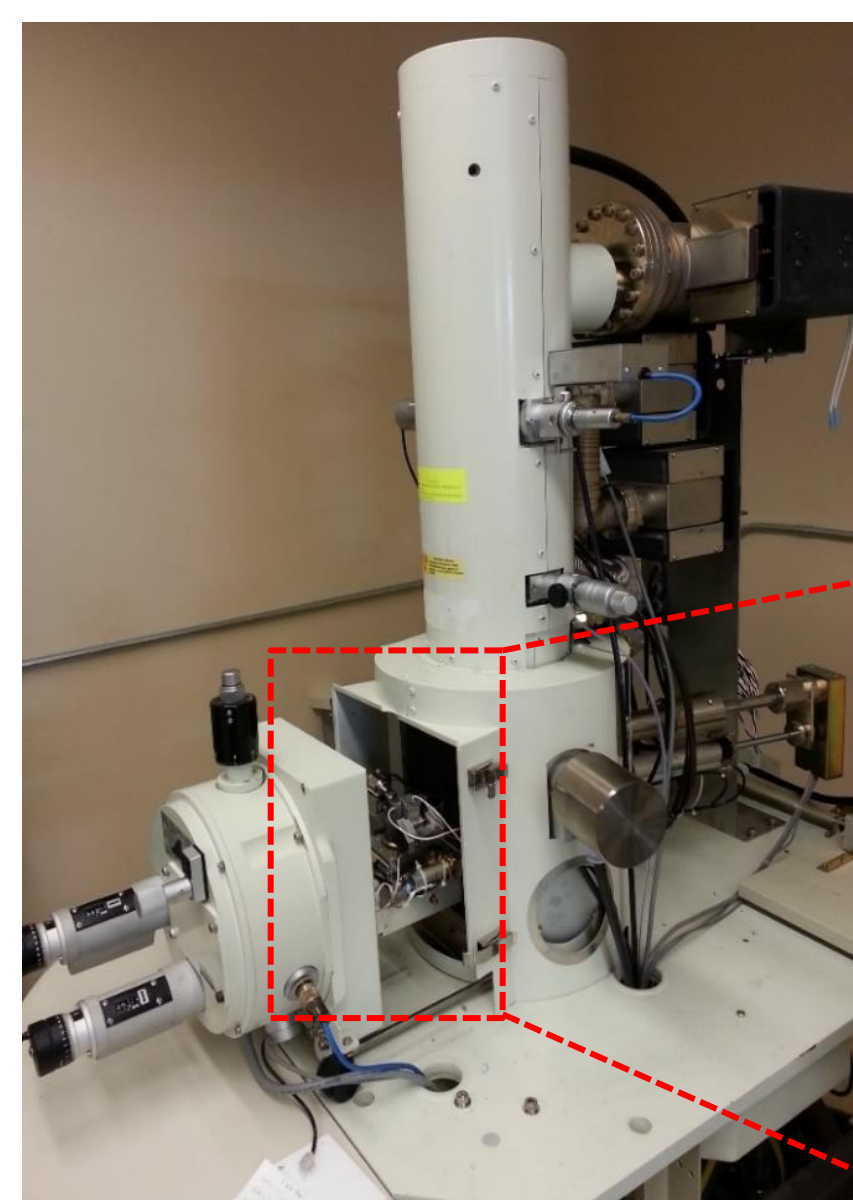
## Micropillar Fabrication and Compression

- Micropillars were fabricated within the interior and boundary regions of the constituent particles.
- Pillars were tested *in-situ* in an SEM using micro-compression.
- Compression data extrapolated to find the yield strength (flow strength at 2% plastic strain).



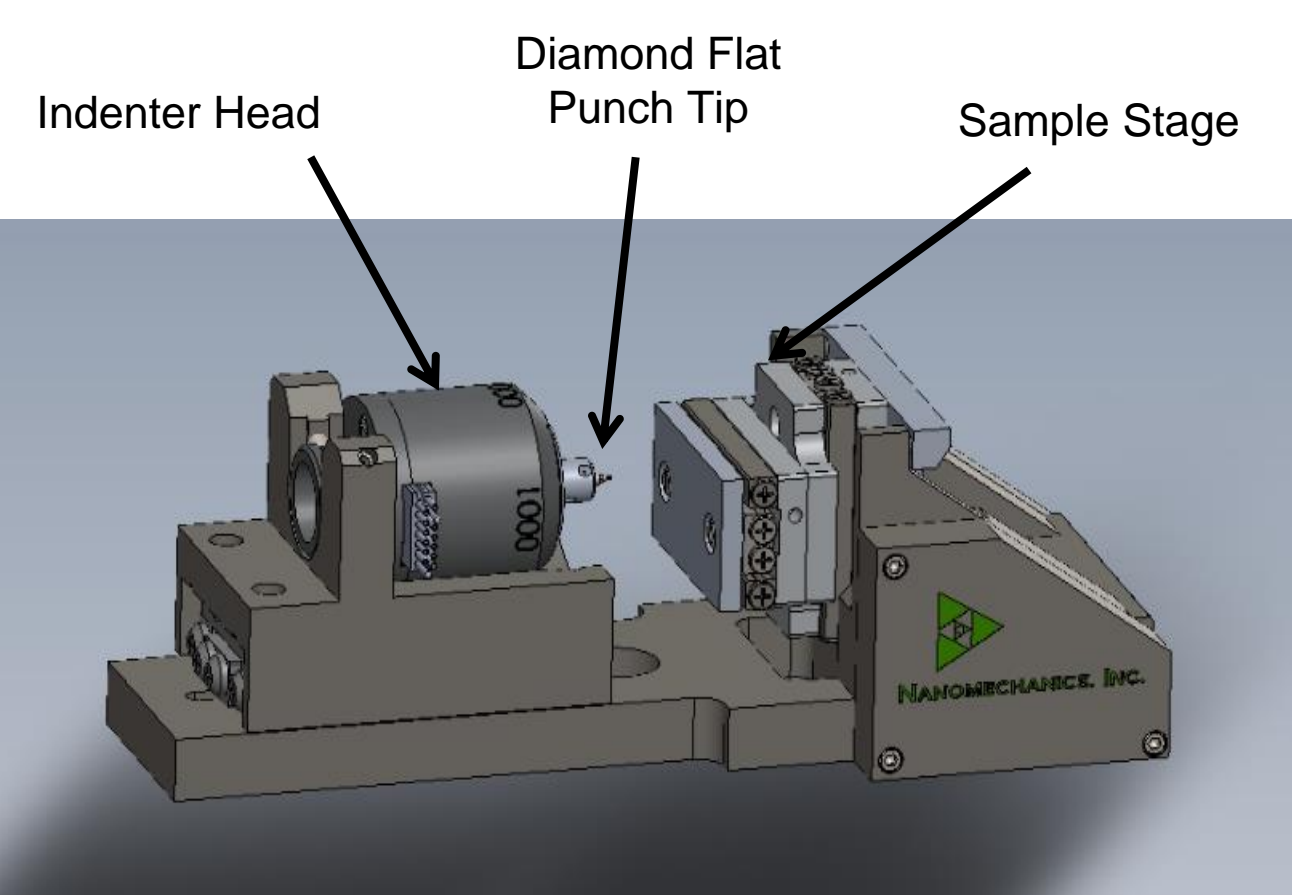
SEM images of micropillars fabricated from the interior and boundary regions

### JEOL 6330F FE SEM

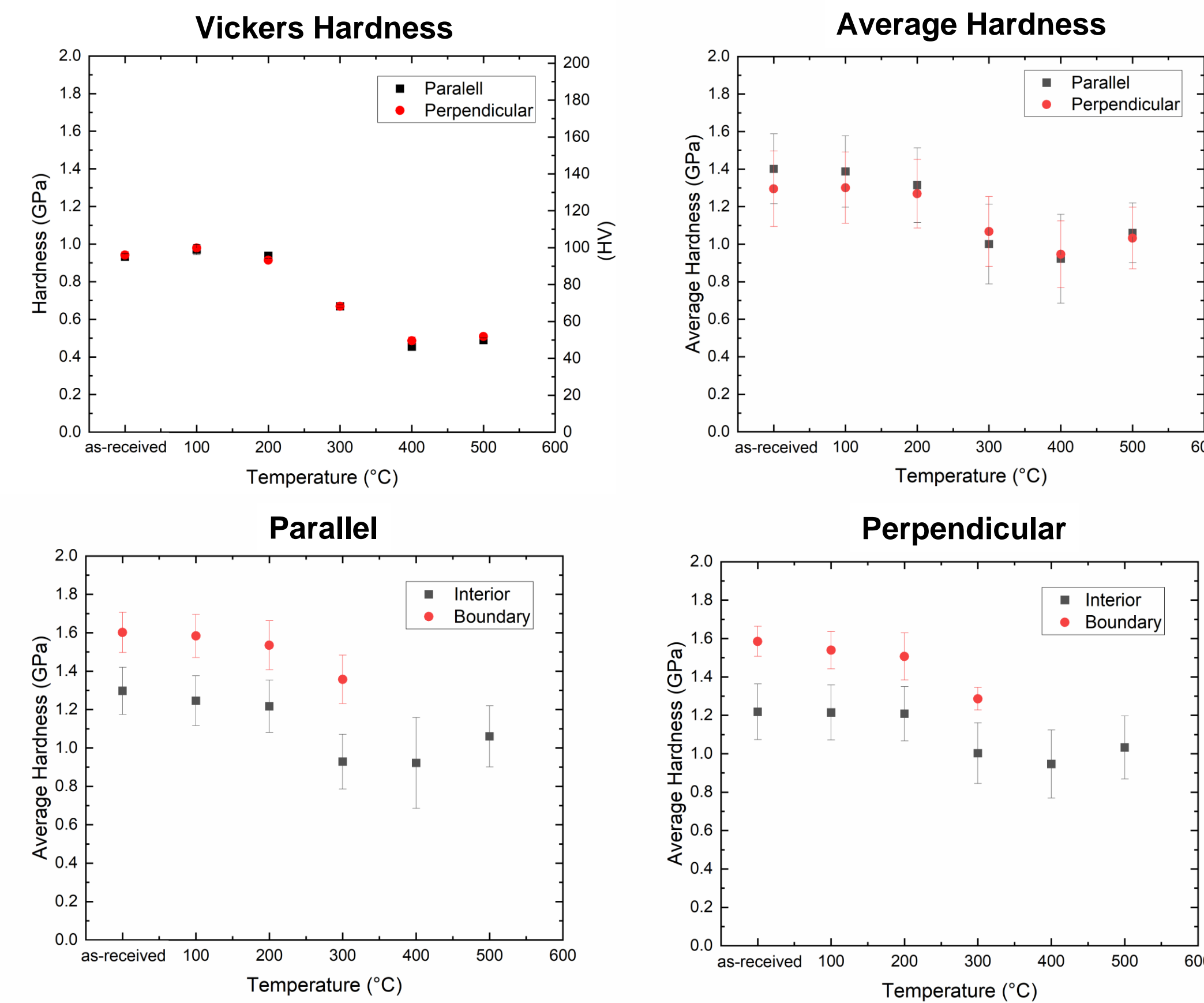


### NanoFlip (KLA)

**Resolution**  
 Displacement: ~ 0.1 Å / Force: ~ 1 nN



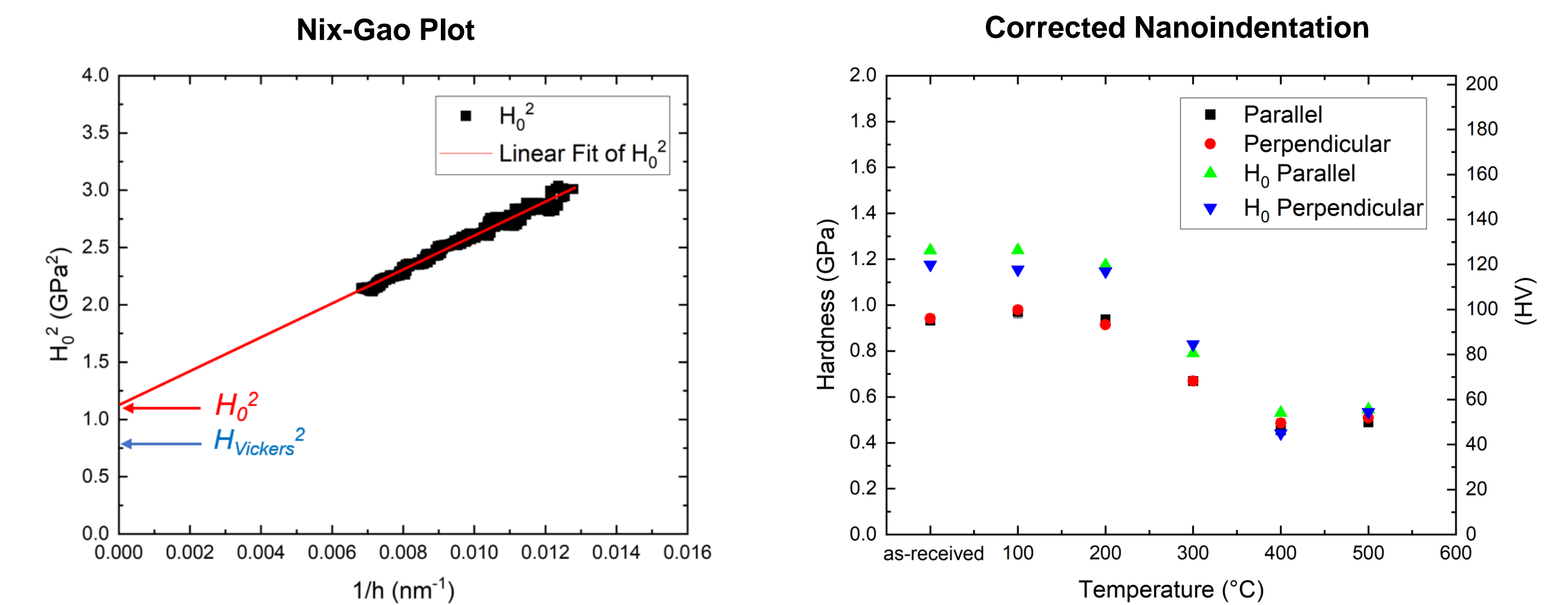
## Nanoindentation



- There is little difference between the parallel and perpendicular directions for both the Vickers and nanoindentation data. There is however a large difference between the interior and boundary regions. This can be attributed to a substantial decrease in the grain size of the boundary region.

## Size Effect Analysis

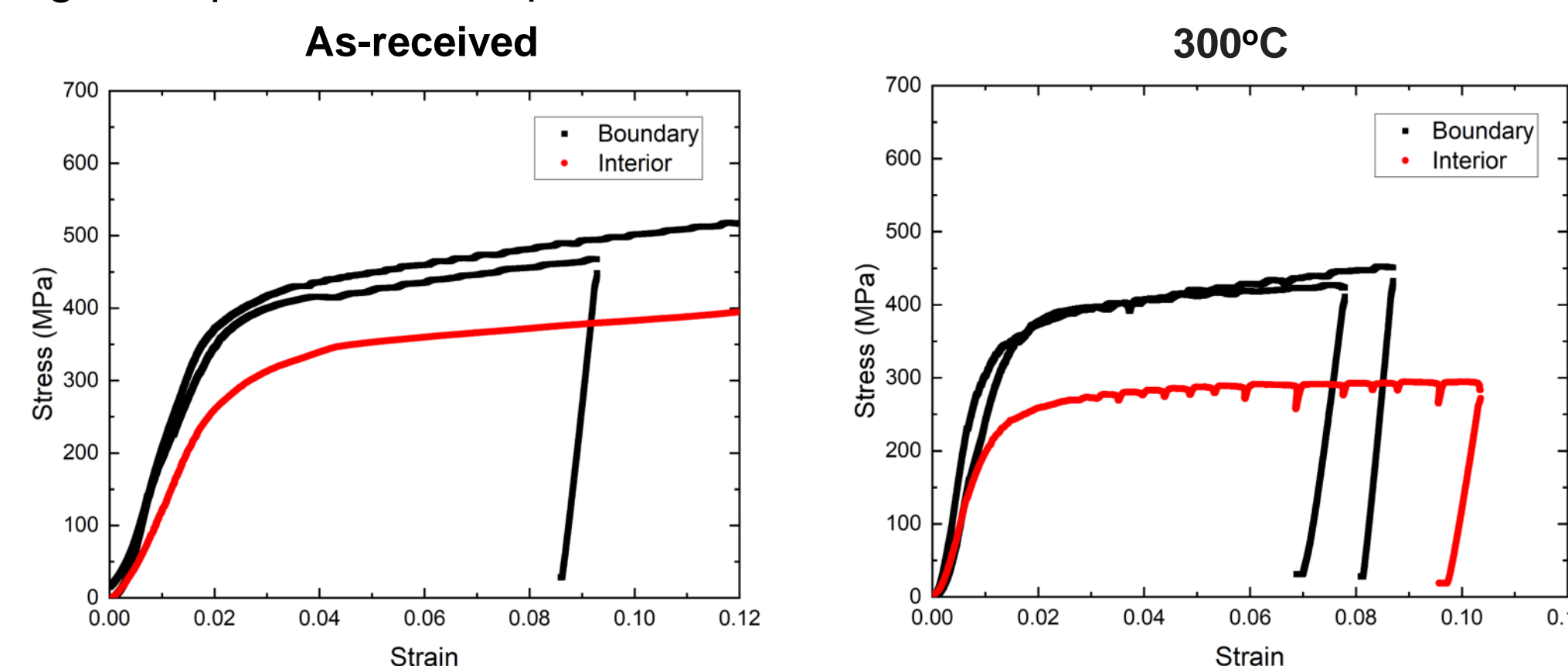
- Note, there is significant difference between Vickers hardness data and the nanoindentation data. Indentation size effect is a well-known phenomena particularly for indentation depths of less than 1 μm.
- The indentation size effect results primarily from geometrically necessary dislocations (GNDs) due to the shape of the indenter tip and can be accounted for using the Nix-Gao Model  $H^2 = H_0^2 + \frac{\alpha}{h}$  where h is the indentation depth, H is the measured hardness  $H_0$  is predicted bulk hardness and  $\alpha$  is a material dependent coefficient.



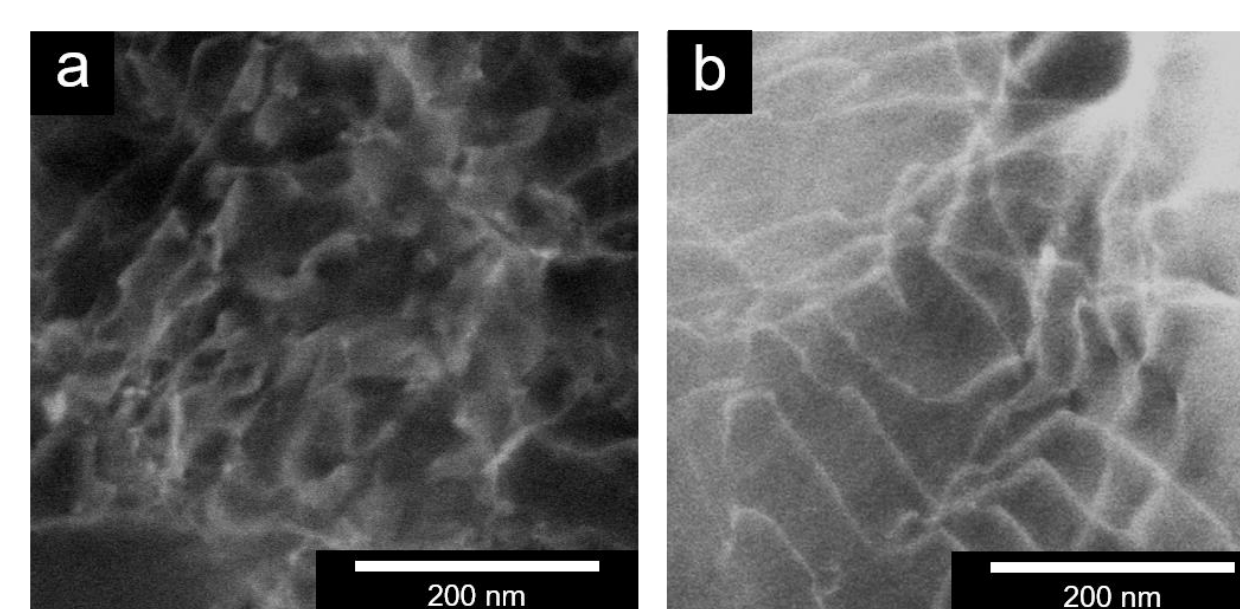
- The remaining difference between the Vickers hardness data and corrected nanoindentation values can be accounted for by intrinsic effects such as grain boundaries and statistically stored dislocations (SSDs).

## Micro-Compression

- Investigation of uniaxial mechanical properties of the coating was performed using micropillars with ~5 μm diameter on the as-received and 300°C sample.



- The as-atomized sample exhibited a substantial difference in yield strength of ~100 MPa. Between the interior and boundary region..
- The reduction in strength between the as-received and 300°C sample can primarily be explained by a reduction in the dislocation density due to thermal annealing.



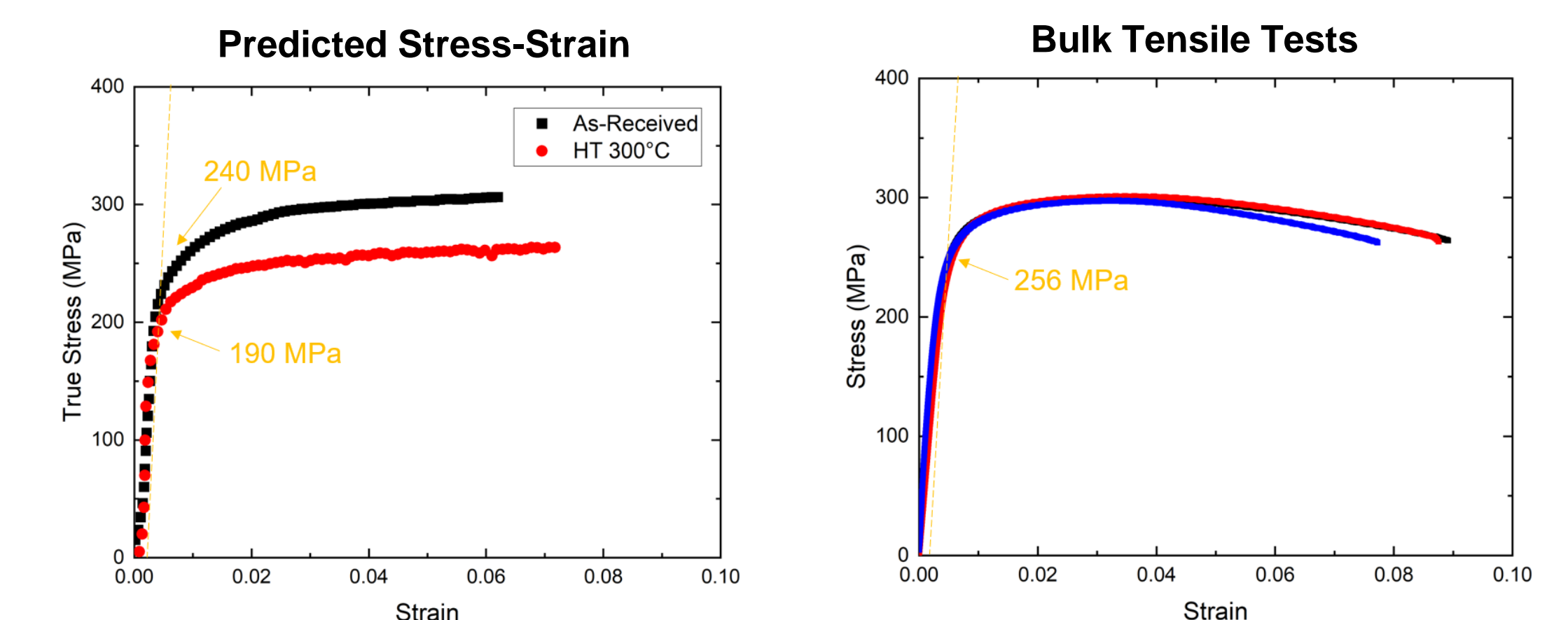
HAADF STEM images of the dislocation structures of (a) as-received and (b) 300°C annealed samples

- TEM imaging reveals a significant reduction in the dislocation density of the interior region  $1.1 \times 10^{15} \text{ m}^{-2} \rightarrow 6.25 \times 10^{14} \text{ m}^{-2}$ .
- From the Taylor equation  $2 \times \tau_y = 2 \times \alpha \mu b \sqrt{\rho}$  we see a difference of ~60 MPa which is in agreement with the experimental results.

## Prediction of bulk stress-strain data

- From our nanoindentation and micropillar experiments it is possible to predict the bulk-scale yield strength by utilizing the rule of mixtures.

$$\sigma_{\text{bulk}} \approx (\sigma_{\text{interior}} V_{\text{interior}} + \sigma_{\text{boundary}} V_{\text{boundary}}) \times \left( \frac{H_{0,\text{nano}}}{H_{\text{Vickers}}} \right)^{-1}$$



- Our predicted value for the yield stress of the as-atomized sample matches well with the actual bulk-scale tension data.

## Conclusion and Future Works

- There is a clear distinction in hardness between the interior regions of the individual constituent particles and the boundary regions which is explained by extreme differences in grain size.
- The unique microstructure of the coating leads to a non-uniform response to heat-treatment. The mechanical data indicated the majority of grain refinement begins to occur at ~300°C with the entirety of constituent particles consumed by a single grain at ~400 °C, after which the microhardness decreases substantially.
- We were able to accurately the macroscopic yield strength of the coating from our micromechanical data. The methodology presented here will be utilized to investigate Ta coldspray systems.