

*Exceptional service in the national interest*



# Characterization of Tri-Lab Tantalum Plate

Thomas E. Buchheit<sup>1</sup>, Ellen K. Cerreta<sup>2</sup>, Lisa Diebler<sup>1</sup>,  
Shuh-Rong Chen<sup>2</sup>, Joseph R. Michael<sup>1</sup> and John Bingert<sup>2</sup>

Materials and Process Science Center  
Sandia National Laboratories  
P.O. Box 5800  
Albuquerque, New Mexico 87185

Materials Science and Technology Division, MST-8  
Los Alamos National Laboratory  
Mail Stop G755  
Los Alamos, New Mexico 87545



U.S. DEPARTMENT OF  
**ENERGY**

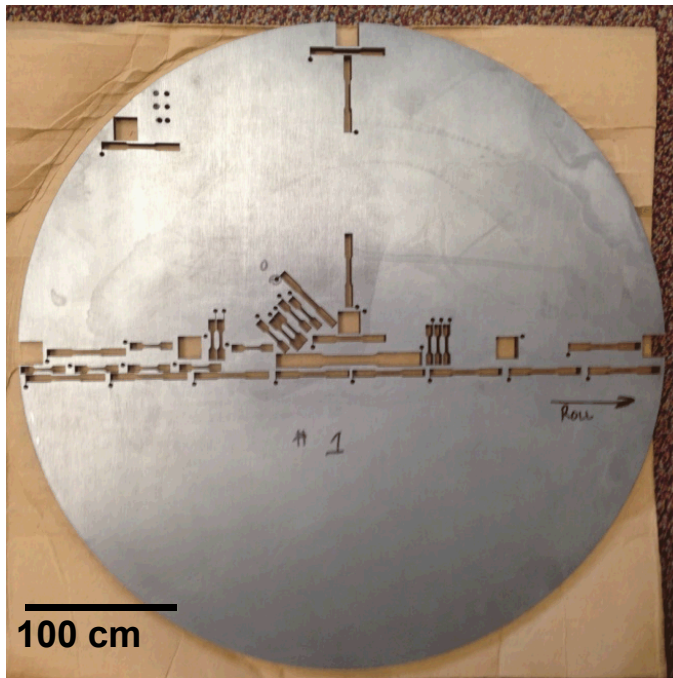


Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND NO. 2011-XXXXP

# Tri-Lab Tantalum plate

-manufactured by HCStark Inc.

*A typical plate:*



0.4 in. thick by 17.5 in. dia.

- 9 plates, 3 to each laboratory (LLNL, LANL, SNL)
- Microstructural banding and crystallographic texture notoriously difficult to control during processing of BCC metals.
- Homogeneity within a plate and plate-to-plate uniformity achieved through a specialized method of clock rolling and tilt rolling of the stock material.

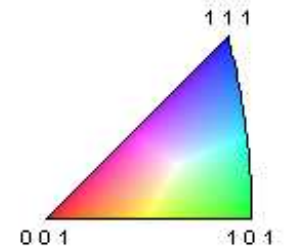
## *Chemistry:*

- Most significant measured impurities are O (16 PPM), N (8 PPM), C (5 PPM) and H (1 PPM).
- Conforms to specification provided by ASTM B-708-05

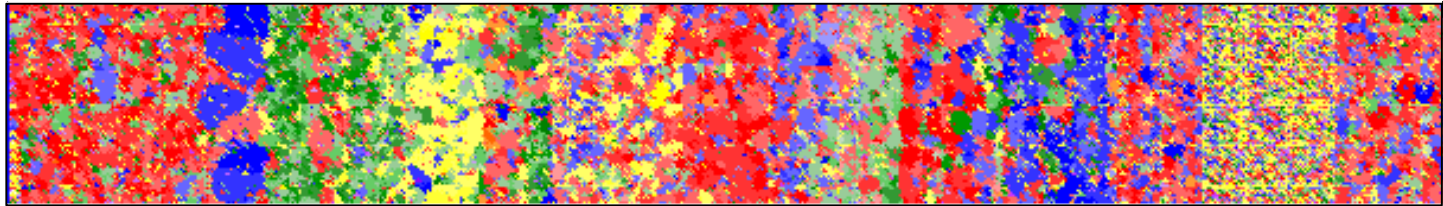
Through thickness and in-plane Microstructure and Mechanical Properties Variation

# Variability in Processed Ta material

Through-Thickness Direction



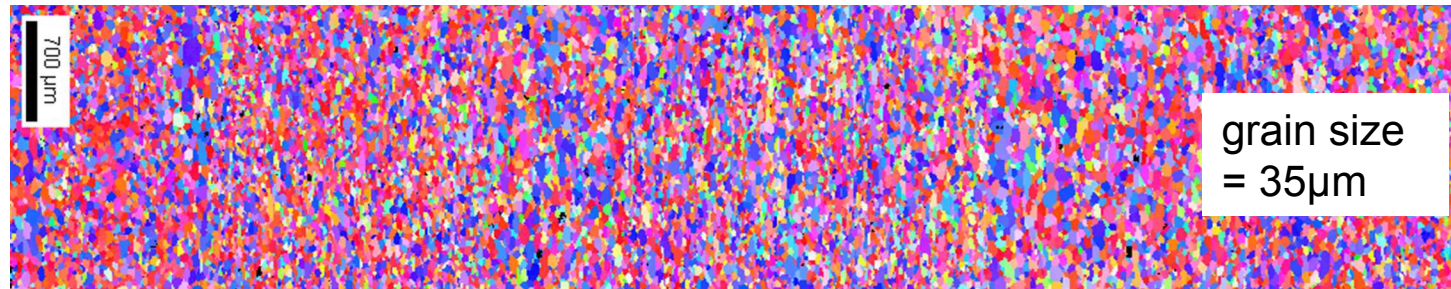
Vintage  
Bar



Previous  
Plate (A)

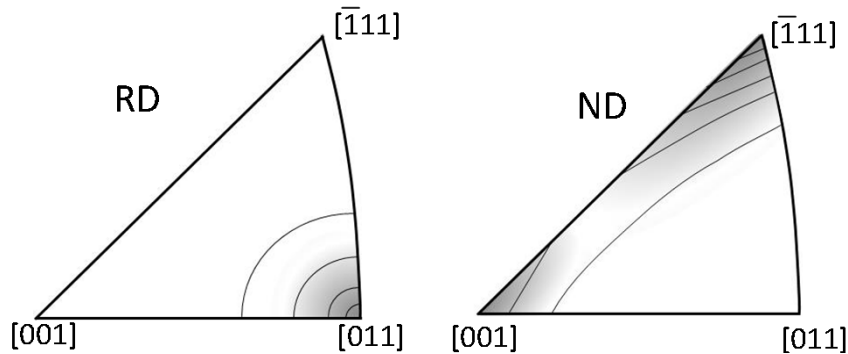


New Plate  
(B)

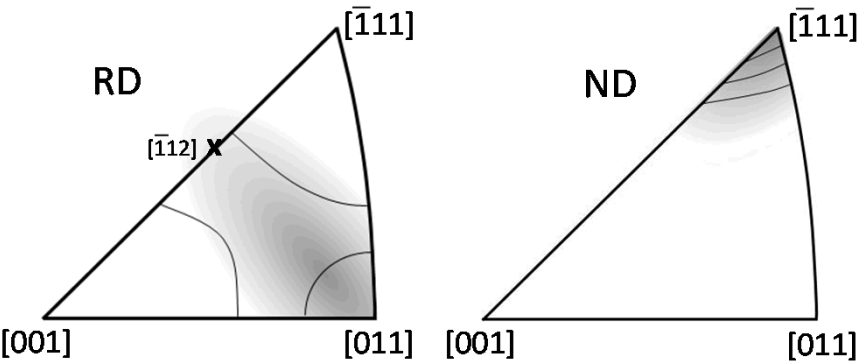


HC Starck Inc.

# Representation of typical rolling textures in BCC metals



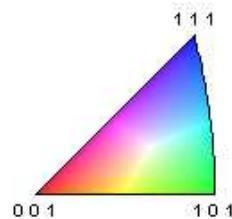
$\langle \gamma \rangle$ - texture



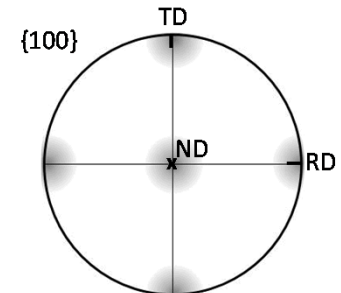
$\gamma$ - texture

$\gamma$ - texture

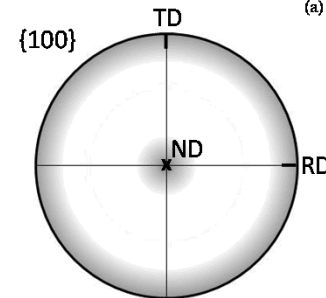
$\langle \gamma \rangle$ - texture



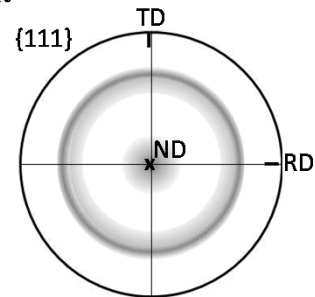
Typical IPF representation of EBSD data



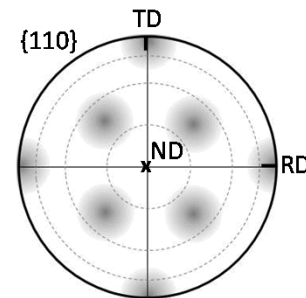
(a) 'cube' texture



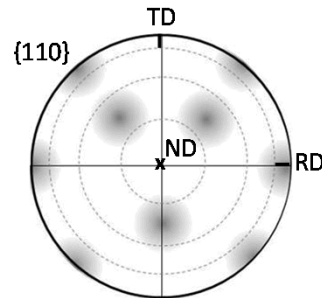
(b)  $\langle 100 \rangle$  fiber texture



(c)  $\langle 111 \rangle$  fiber texture



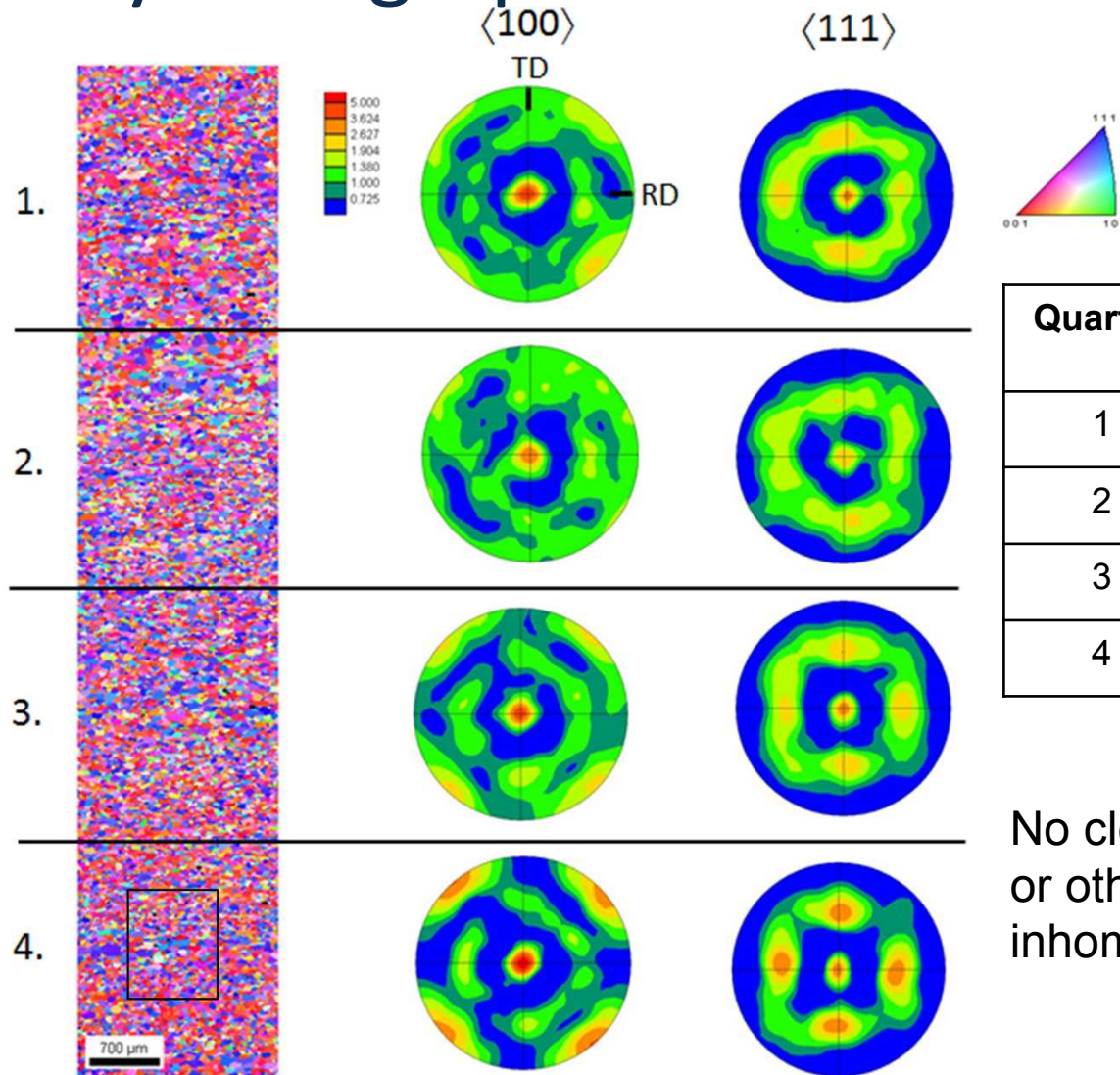
(d)  $\{110\}[011]$  rolling texture



(e)  $\{111\}[011]$  rolling texture



# The $\alpha$ - fiber component characterizes the crystallographic texture of the plate



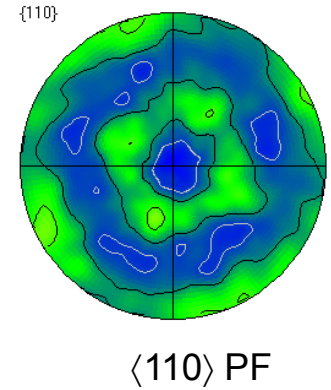
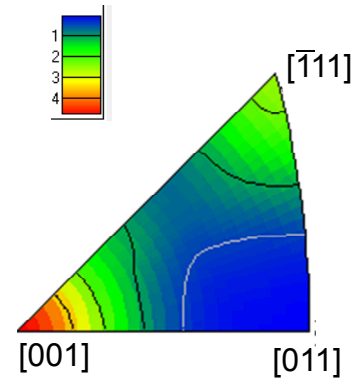
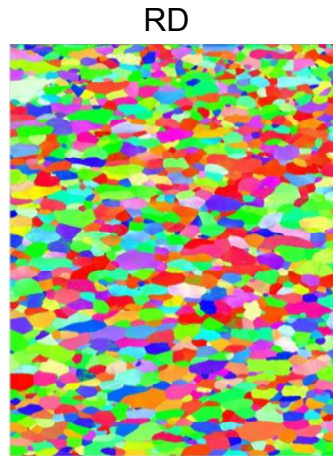
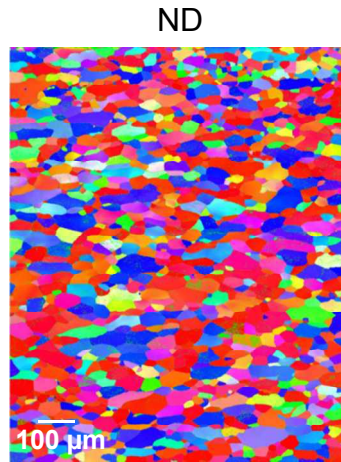
Quartile	$\langle 001 \rangle / \langle 111 \rangle$ ratio	Grain Size ( $\mu\text{m}$ )
1	1.24	38
2	0.957	36
3	1.05	35
4	1.39	34

No clear evidence of banding or other microstructure inhomogeneity

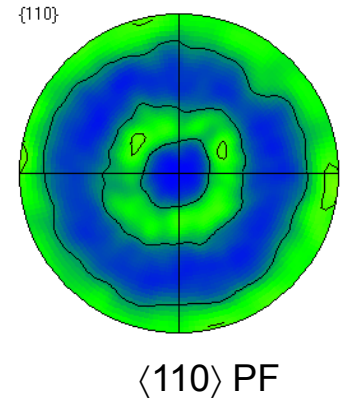
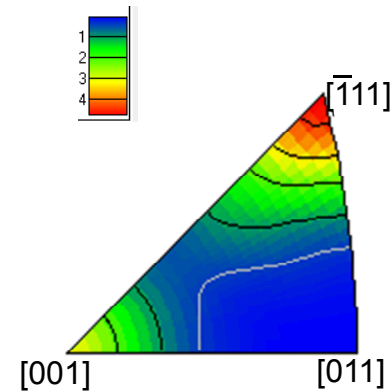
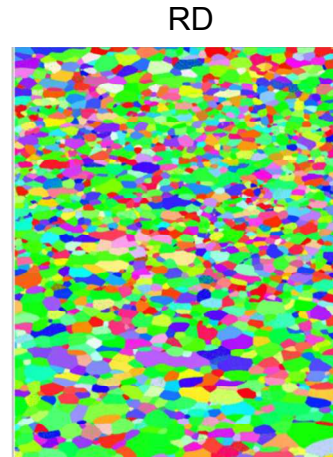
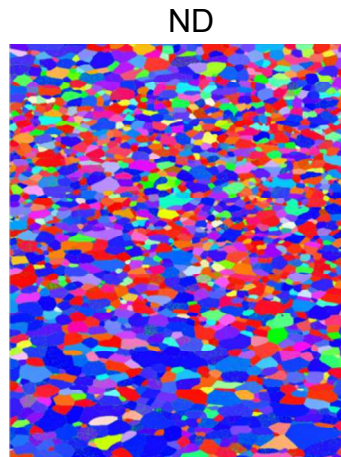
# High Resolution EBSD maps from the center of the plate

1  $\mu\text{m}$  pixel spacing

Near plate surface



Near plate center

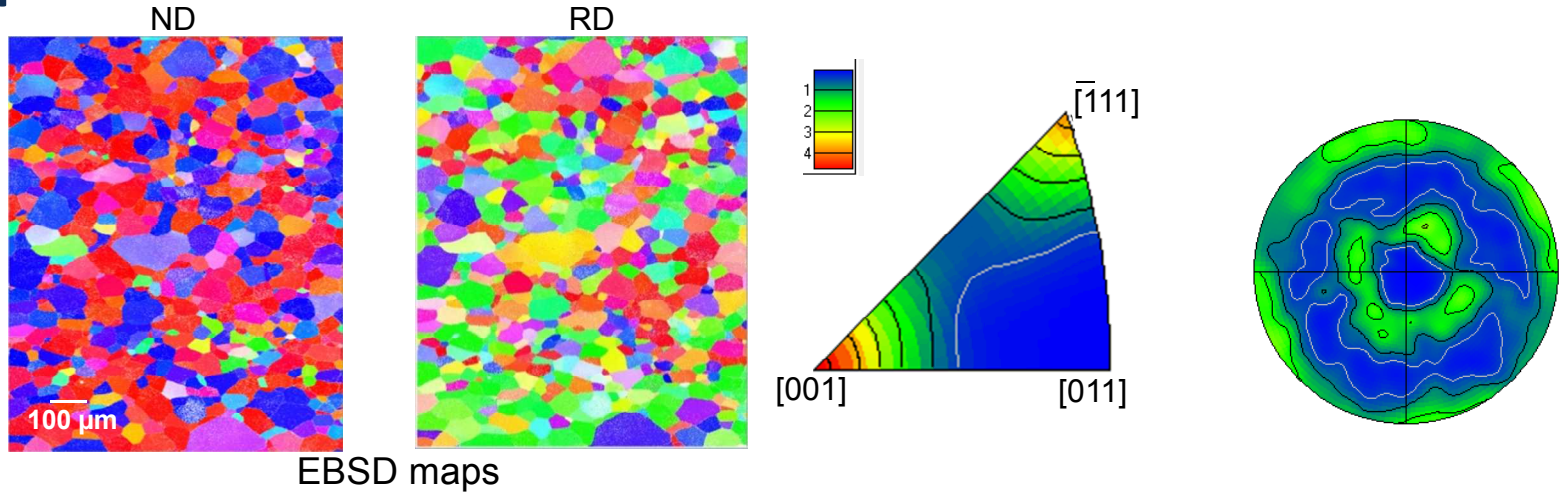


- Partial  $\langle 110 \rangle$  fiber texture along RD and  $\langle 001 \rangle / \langle 111 \rangle$  ratio changes from center to surface along ND

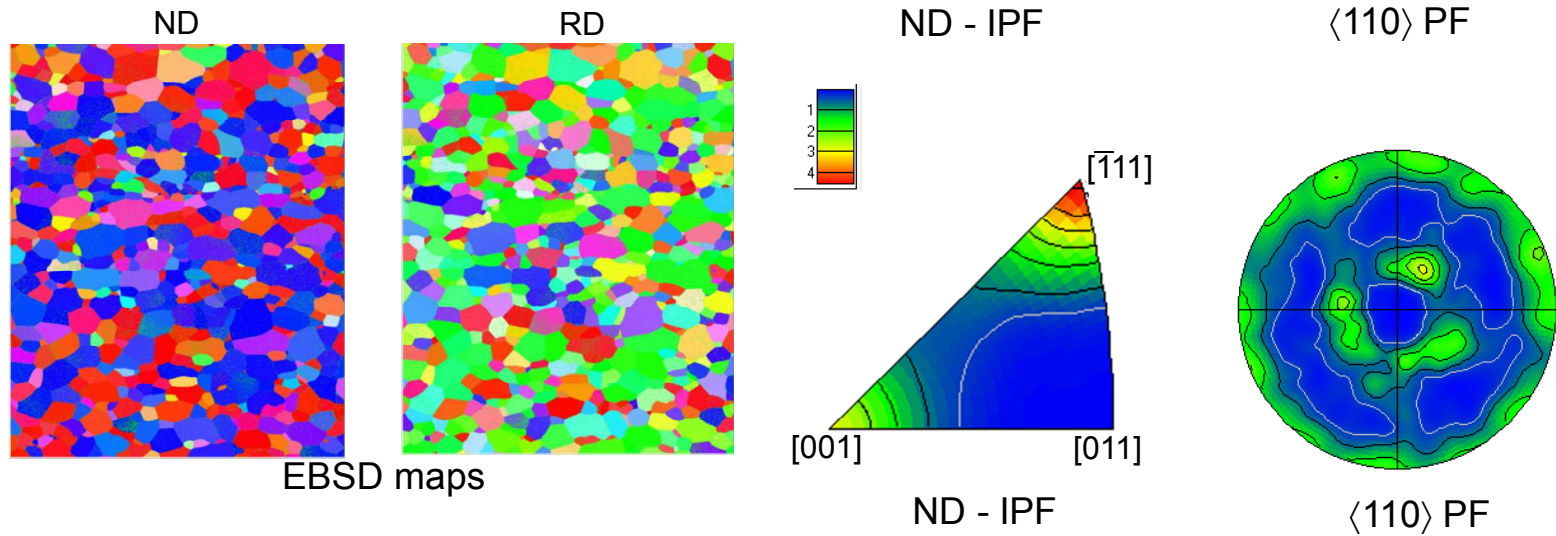


# High Resolution EBSD scans from the end of the plate

**Near plate surface**



**Near plate center**



- Increased grain size near end of plate

# Summary Tables from EBSD maps

Through Thickness scan

Quartile	$\langle 001 \rangle / \langle 111 \rangle$ ratio	Grain Size ( $\mu\text{m}$ )
1	1.24	38
2	0.957	36
3	1.05	35
4	1.39	34

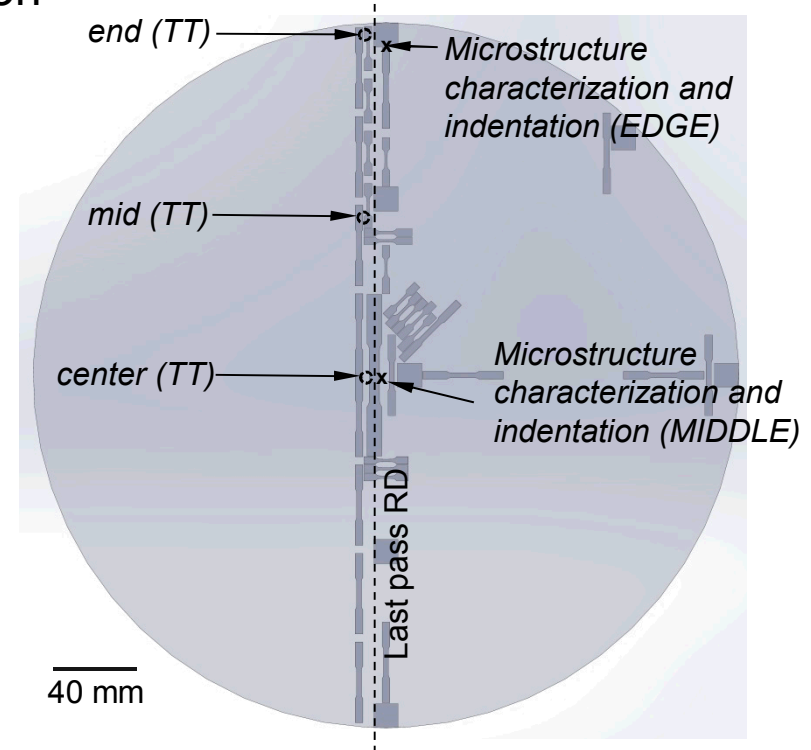
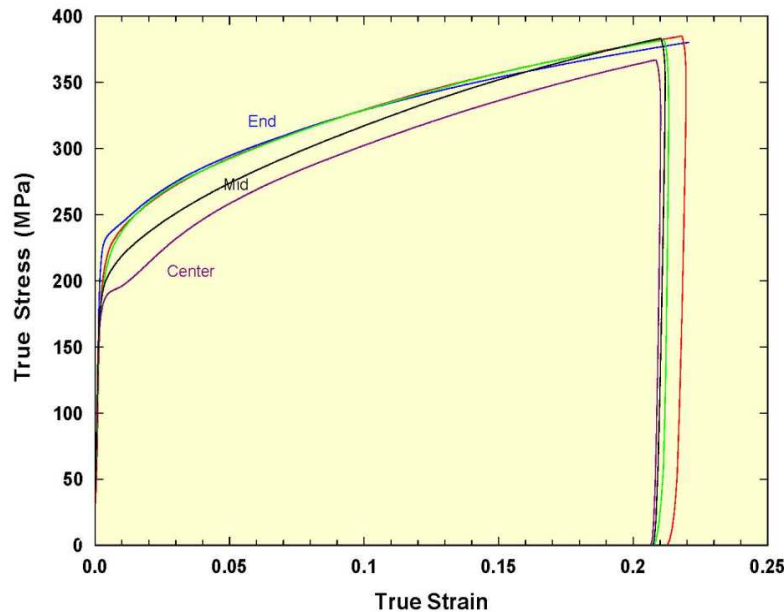
High Resolution scans

Location	$\langle 001 \rangle / \langle 111 \rangle$ ratio	Grain Size ( $\mu\text{m}$ )	Aspect Ratio
Center Top Edge	3.99/2.17 (1.83)	27.1	1.63
Center Middle	2.89/4.29 (0.67)	23.7	1.60
End Top Edge	5.16/4.07 (1.26)	38.3	1.12
End Middle	3.87/6.00 (0.64)	39.7	1.07



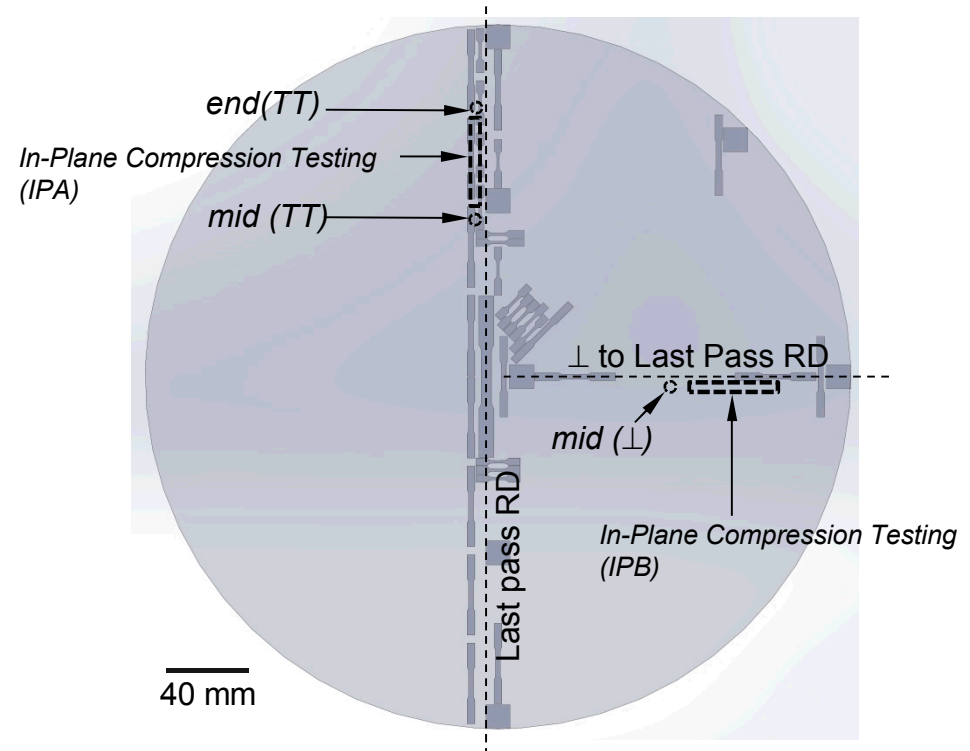
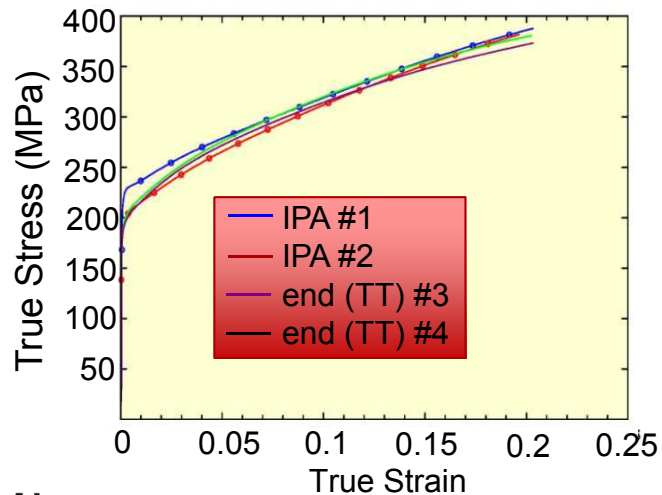
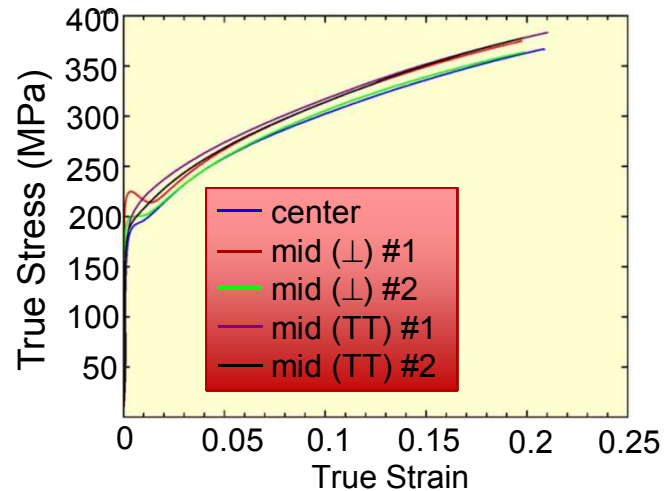
# TT Compression Testing suggested a trend of increasing strength from center to end of plate

- Along the last pass rolling direction



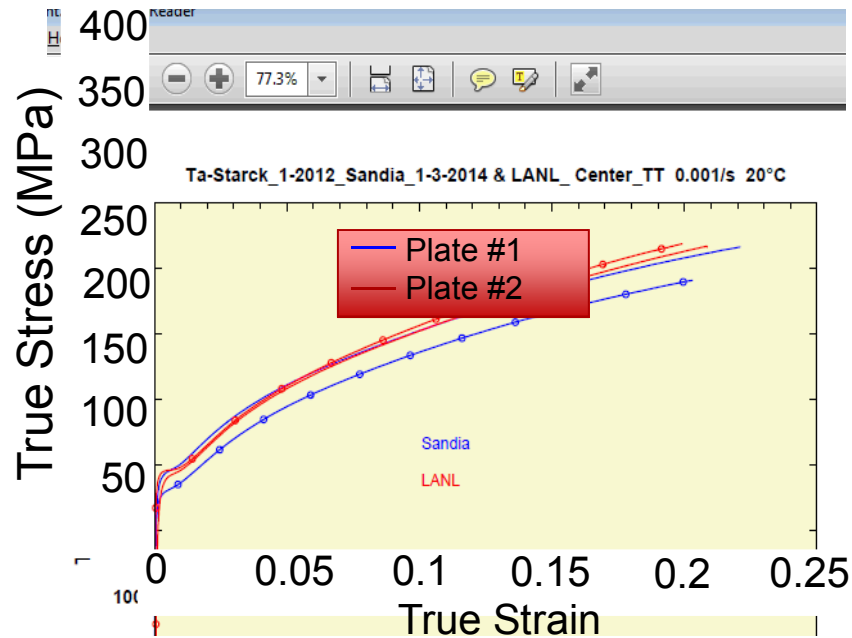
- Compression test Cylinders: 5mm dia. X 5 mm height
- Center/end difference consistent to a strain of 1.1
- Rationalized in terms of texture variation

# Compression testing suggested minimal directional and in-plane variation in mechanical properties



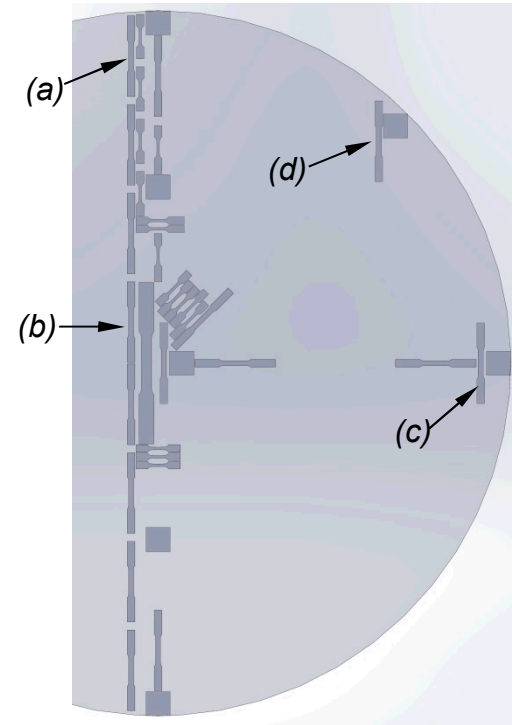
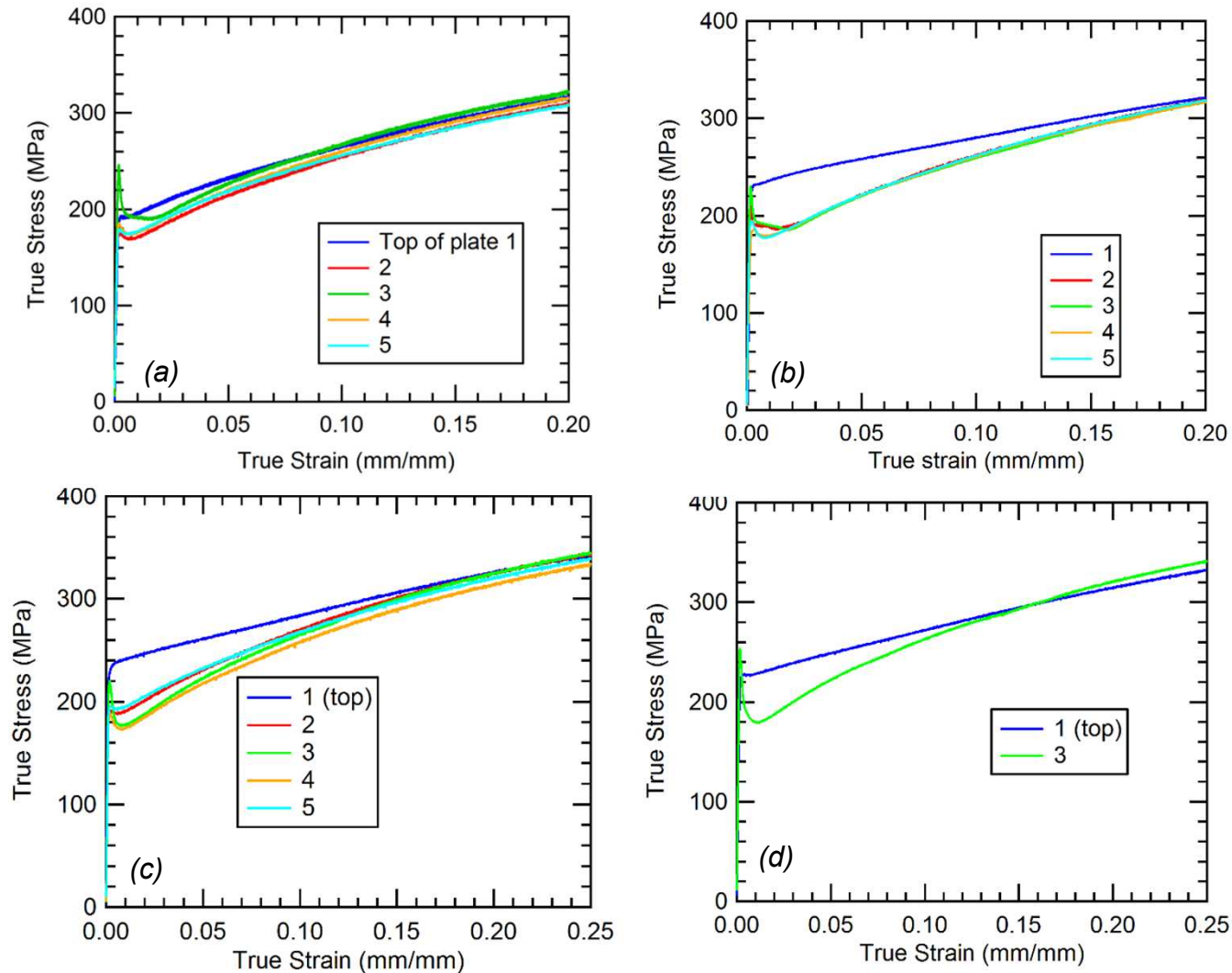
- Slightly different work hardening response in IPA compression test results

# Negligible Plate-to-Plate variation demonstrated



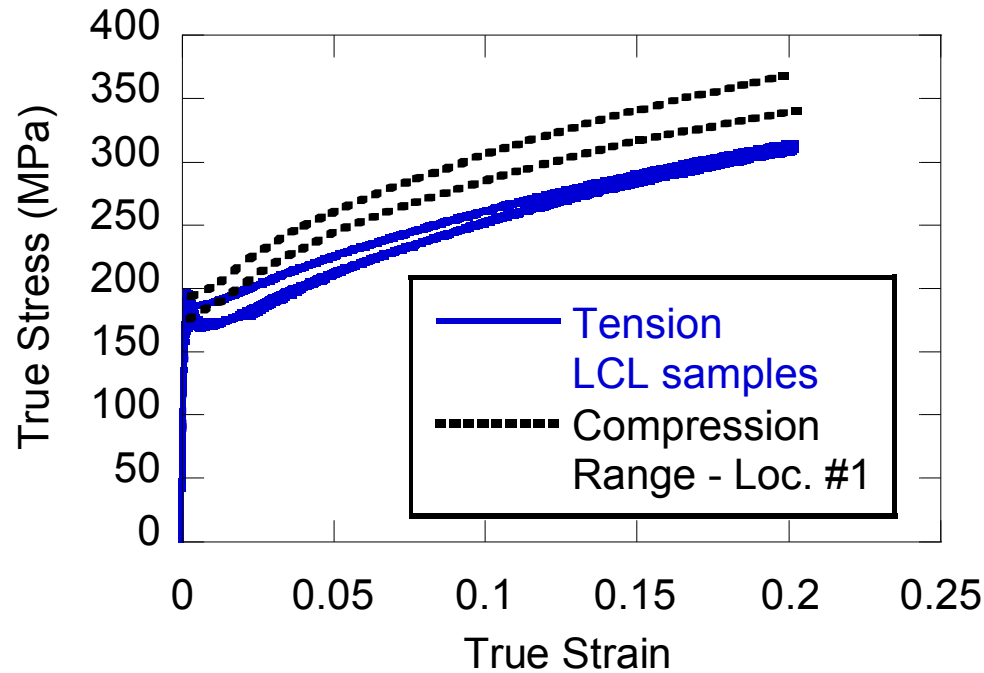


# Stored work near the plate surface suppressed yield point effects



ASTM 1/2 sub-size:  
thickness = 0.063 in.

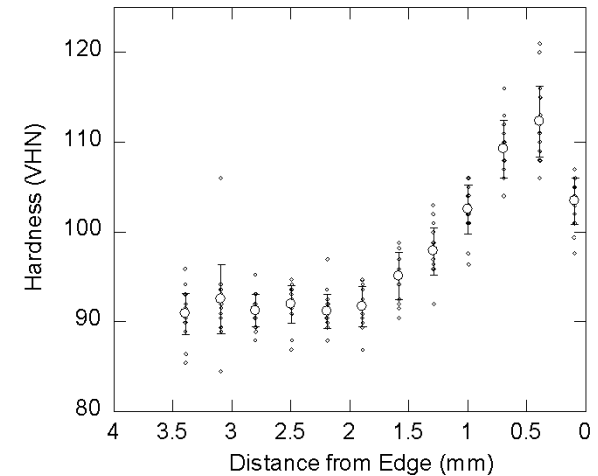
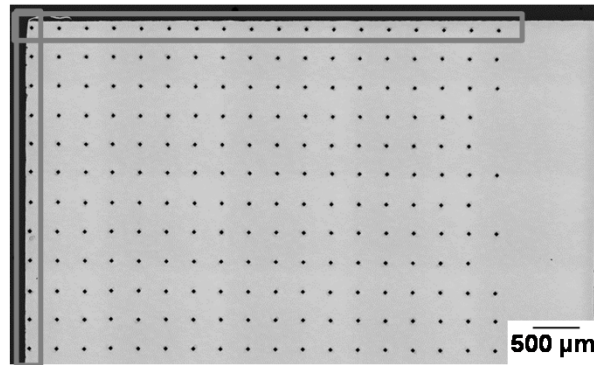
# Work hardening rates differed in compression tests vs. tension tests



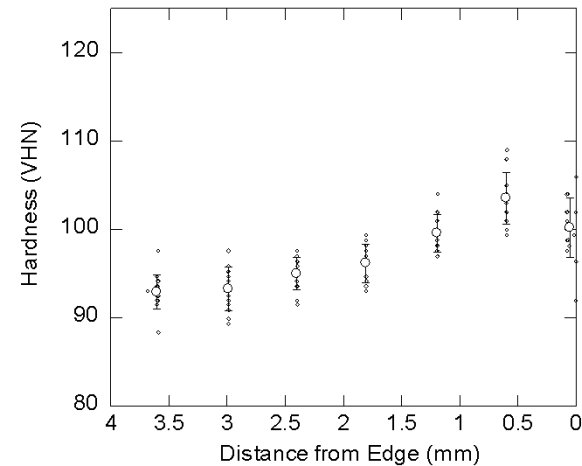
- Tension compression asymmetry in BCC metals
- Different geometry of test samples
- Orientation hardening

# Micro-Indentation showed increased hardness near the surface of the plate

center of plate



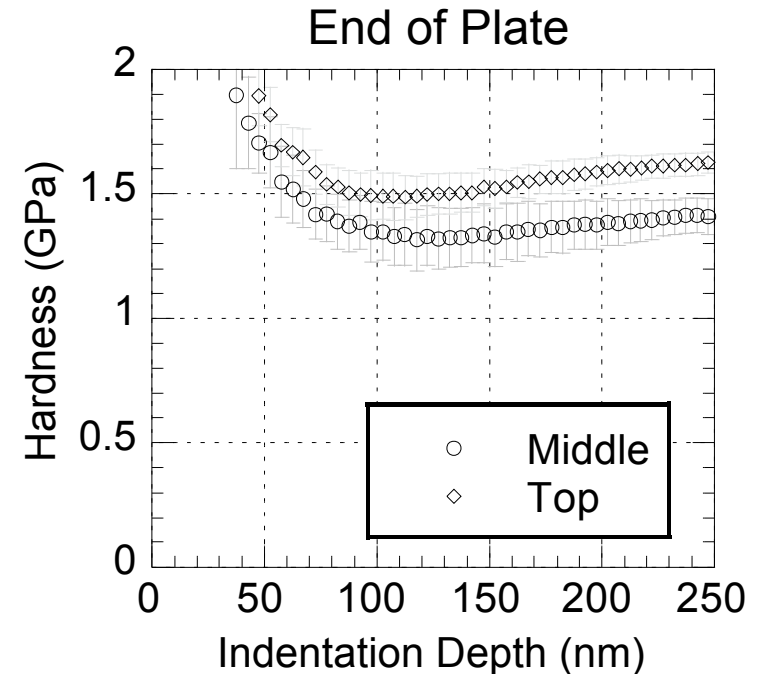
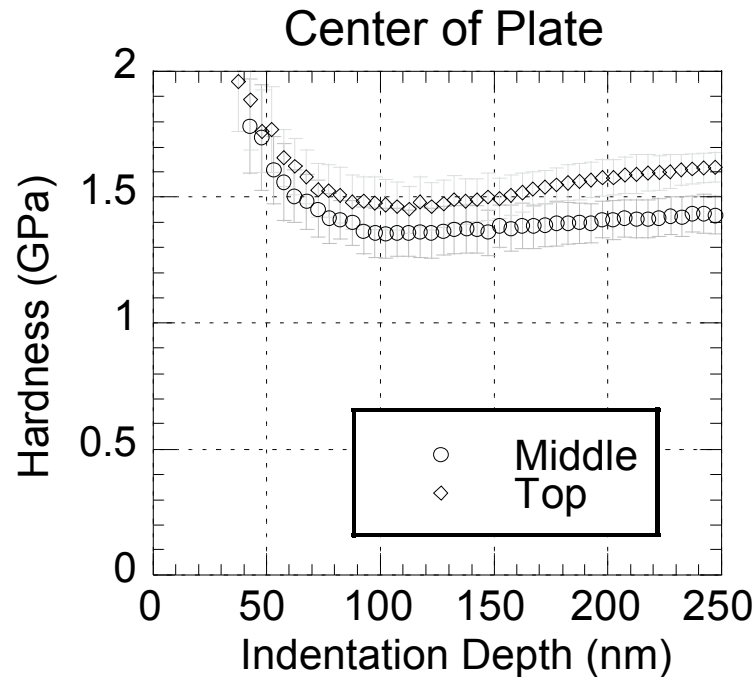
end of plate



Evidence of stored work near top of plate



# Instrumented Indentation reveals similar 15% increase in hardness near edge of plate



Different scale experiment vs. microindentation

# Summary

- DOE Ta has uniform crystallographic texture, far superior that what can usually be achieved for a polycrystalline BCC metal. Variation in  $\langle 001 \rangle / \langle 111 \rangle$  ratio near end of plate.
- Evidence of stored work near top and bottom surfaces of the plate and a small increase in grain size (from 25  $\mu\text{m}$  to 39  $\mu\text{m}$ ) near the end of the plate.
- No clear evidence of microstructure banding.
- Compression cylinder testing revealed a measurable increase in strength near the end of the plate.

**Recommendation: Do not use material 25 mm from the end of the plate and 2mm from the top and bottom of the plate for ACP testing**