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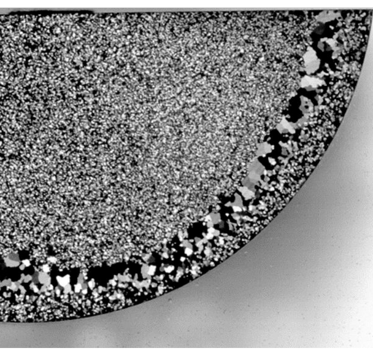

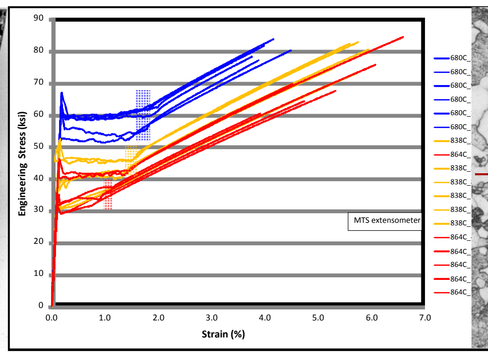

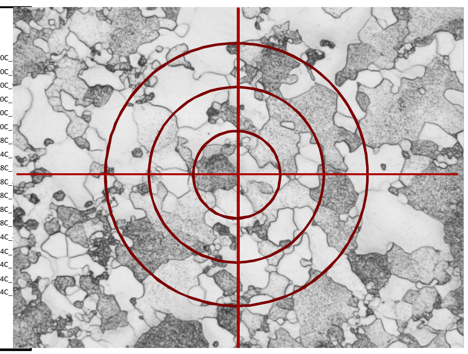

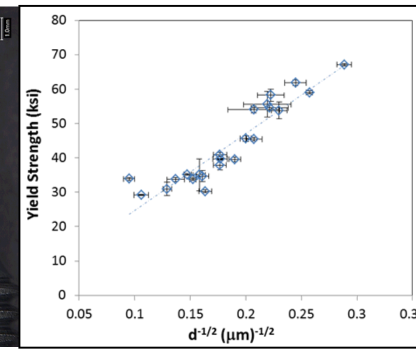

Hall-Petch Behavior of Fe-Co-V Soft Magnetic Alloy Barstock

Don Susan, Tom Crenshaw, Jeff Rodelas, Charlie Robino, and Bill Greenwood

Sandia National Laboratories, Albuquerque, NM

Materials Science and Technology 2014, Pittsburgh, PA

October 13, 2014

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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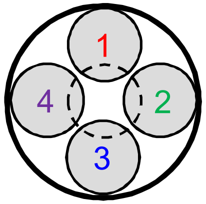
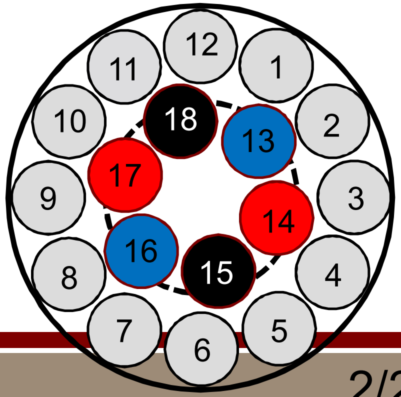
Motivation

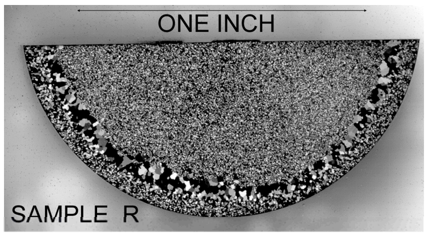
- **Hiperco® 50A, also known as 2V Permendur: Best soft magnetic properties of any alloy. *But*, low strength with wide scatter, and low ductility. Hypothesis: Non-uniform grain size in bar product, and sometimes very large grains (abnormal grain growth), are causing the low strength and high variability. (®Hiperco is a registered trademark of Carpenter Technology Corporation.)**
- **The goals of this work were to 1) measure grain size, 2) correlate strength to grain size in Hiperco®, specifically <u>bar product</u>, 3) make initial correlations of structure and mechanical properties to magnetic behavior.**
- **Note: "Modified" Hiperco® 50A, 500 ppm Nb added**
  - **49Fe-49Co-2V + 500 ppm Nb**
  - **Grain size measured across bar diameter for 2-inch and 1-inch diam. bars.**
  - **Tensile bars were removed from outside and "inside" (2 inch bar) and outside and center of 1-inch bar.**
  - **Range of heat treatments employed:**
    - **864C, 2hrs, 838C, 1hr and 2hrs, 754C, 720C, 1hr and 2hrs, 680C, 2hrs**
  - **Heat treat optimizes *magnetic performance***



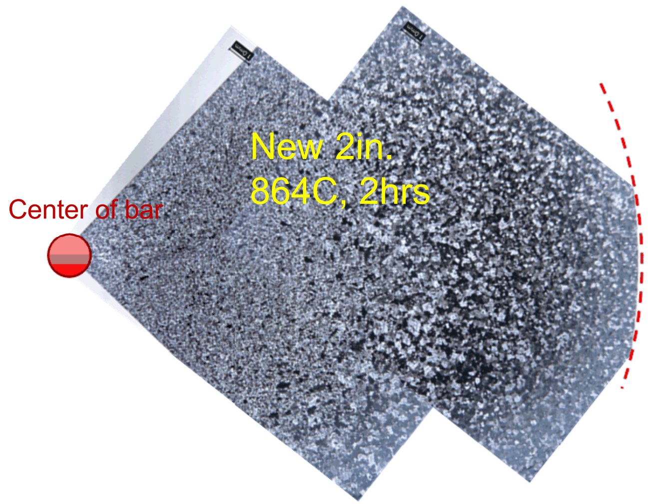
Tensile bar locations



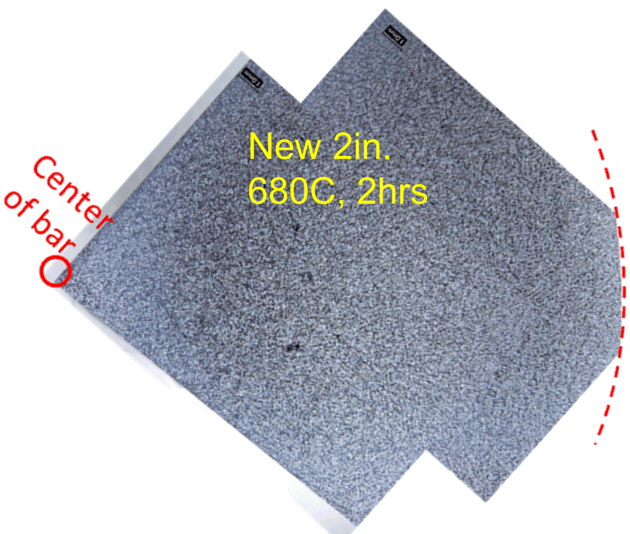
Qualitative Observations of Non-Uniform Grain Size in Heat Treated Bar

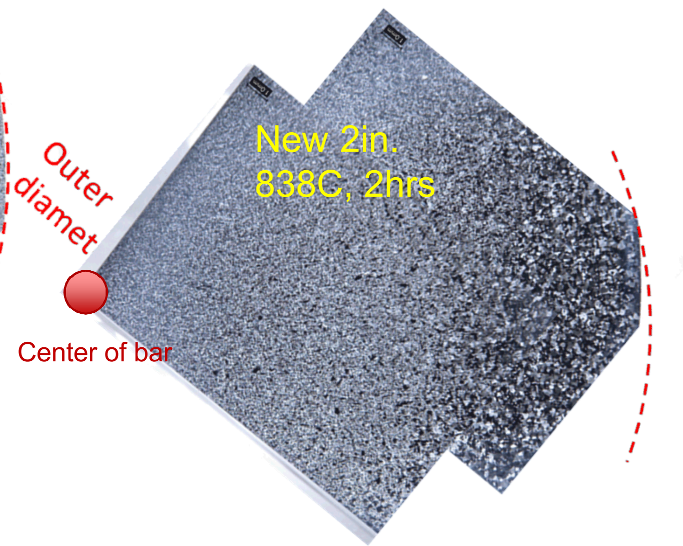


Historic (~2008) Hiperco® bar



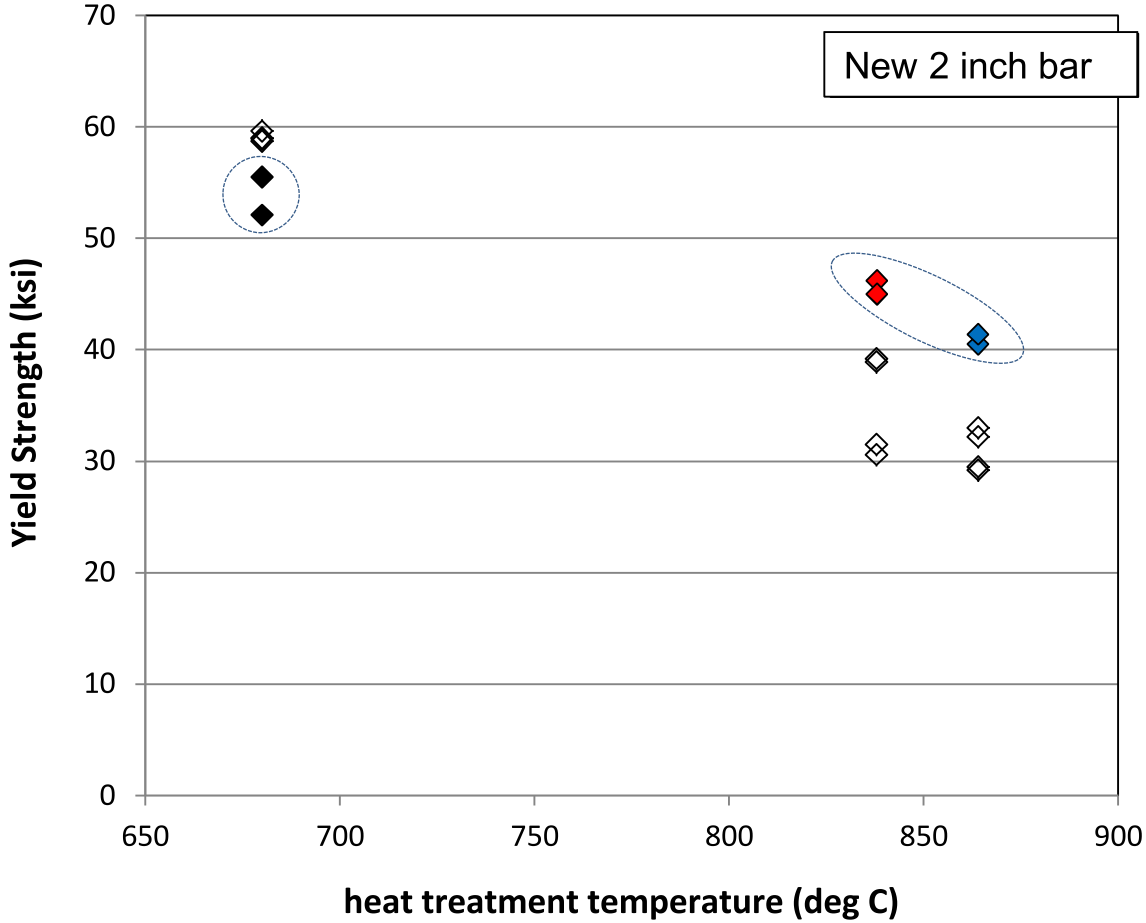
New 1in.
838C, 2hrs

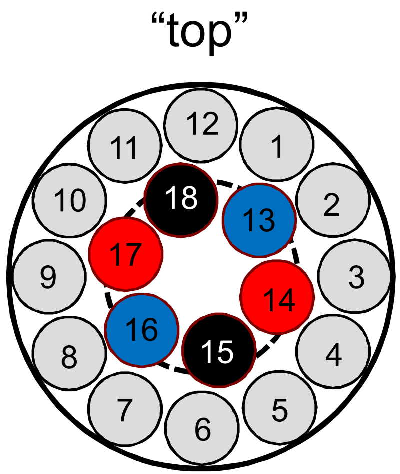




New 1in.
720C, 2hrs

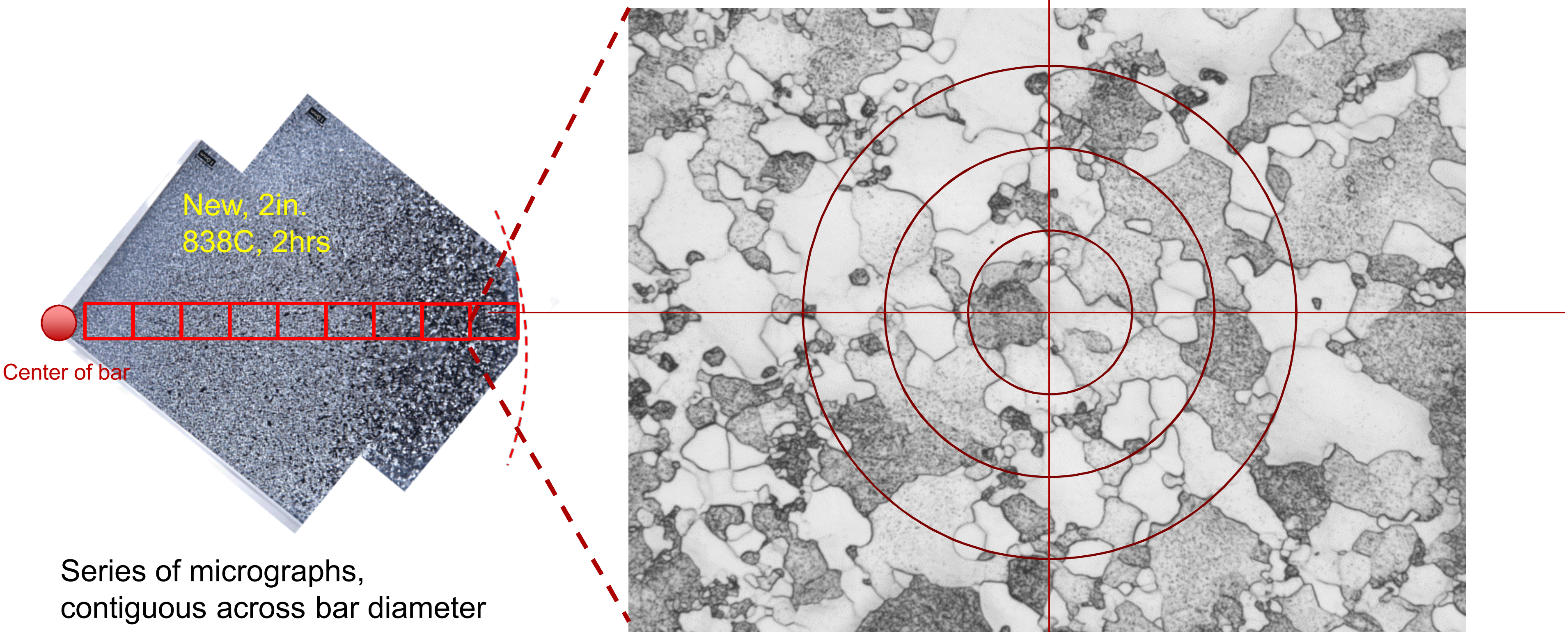
Tensile Test Results from “New” 2-inch diam. bar





Grain Size Measurement Technique

- Modified Abrams 3-Circle Method, ASTM E112-12
- Up to 22 individual micrographs were analyzed per sample, depending on magnification and bar diameter



Series of micrographs, contiguous across bar diameter

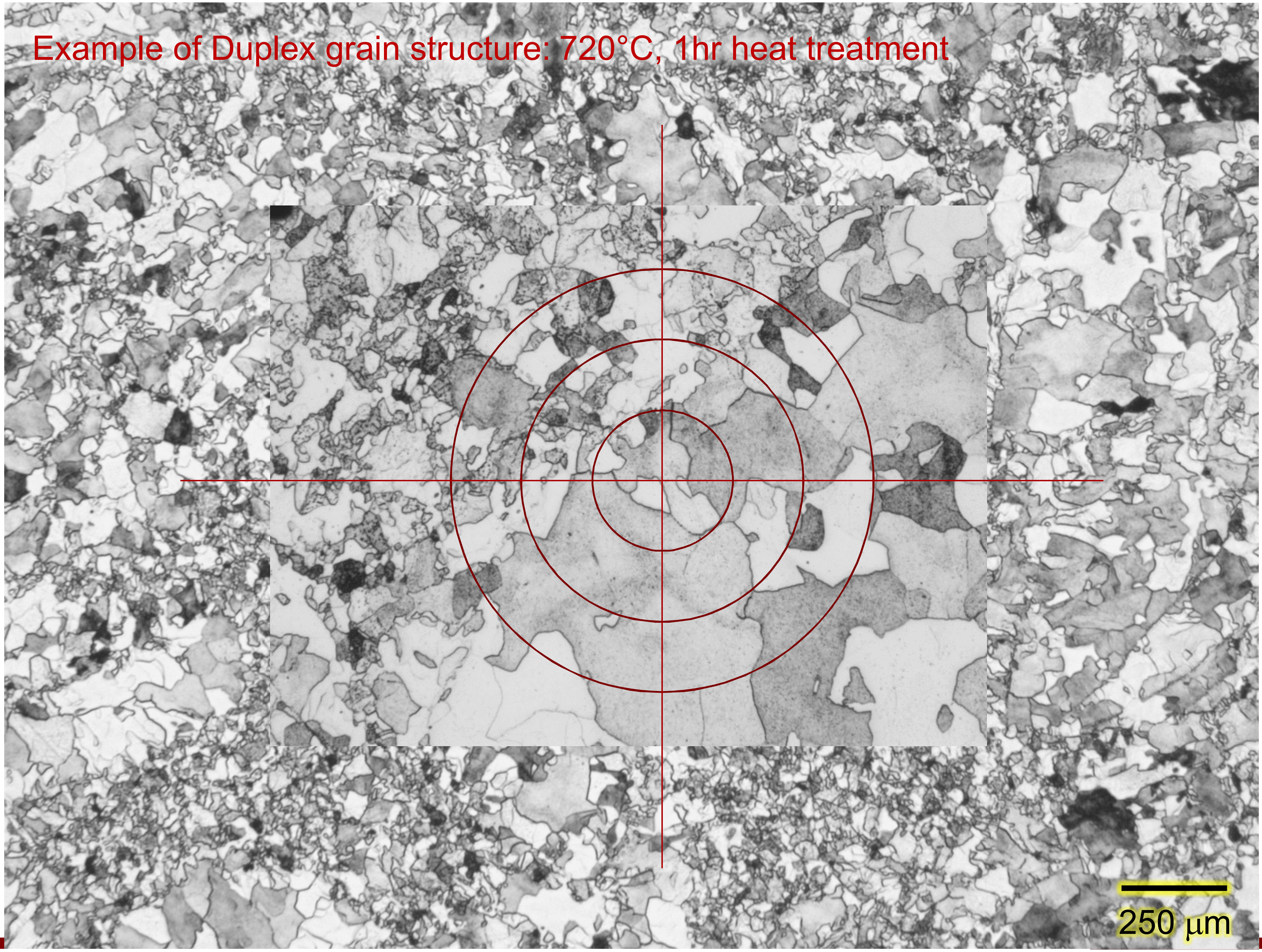
$$\overline{N}_L = \frac{N_i}{L/M}$$

$$\overline{l} = 1/\overline{N}_L$$

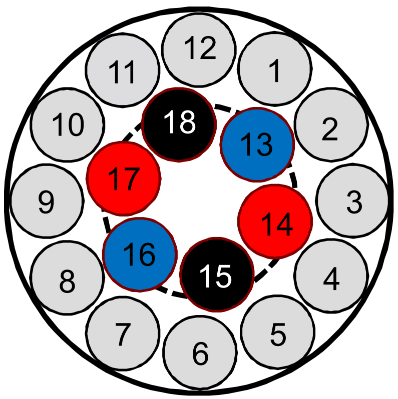
Sources of Data Scatter

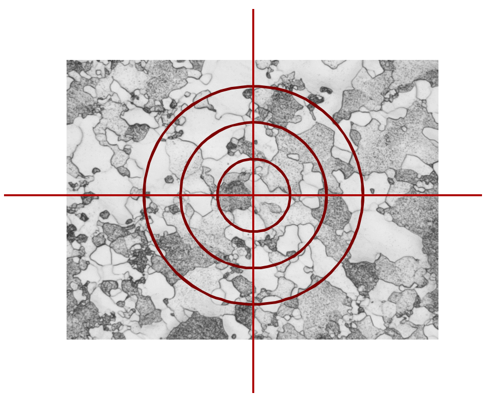
- Duplex grain structure, especially in transition zone from outside to center of bar
- Tensile bars, especially "outside samples", contain a gradient of grain sizes
- Incomplete delineation of all grain boundaries by the chemical etch. Etchant is Petzow Com3: 100 ml DI H_2O, 100 ml HCl (37%), 200 ml methanol, 5 ml HNO_3 (69%), 7g $FeCl_3$, 2g $CuCl_2$…immerse 10-15 seconds
- Counting errors
- Other sample preparation or etching effects

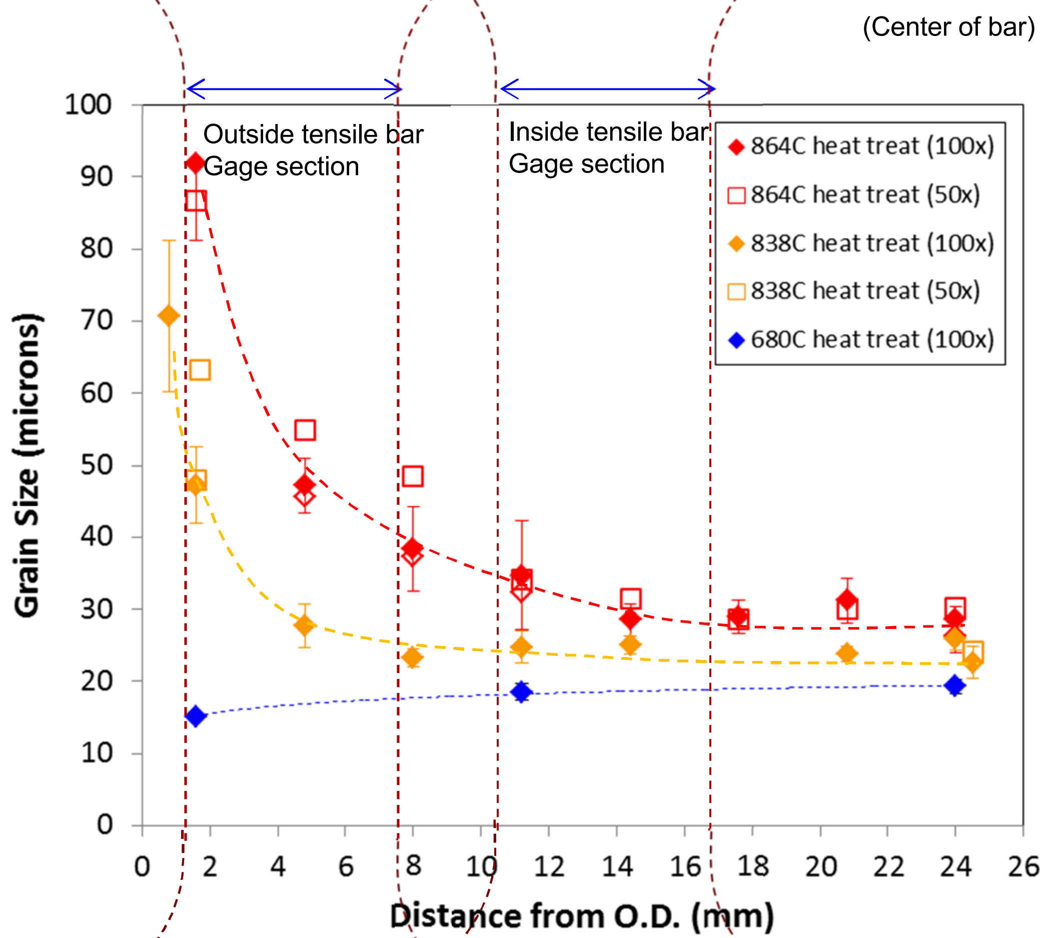
Example of Duplex grain structure: 720°C, 1hr heat treatment

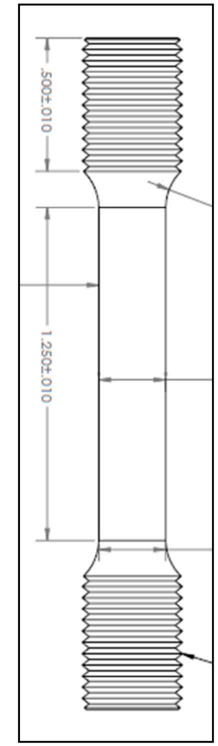


Grain Size of 2-inch Diam. Bar



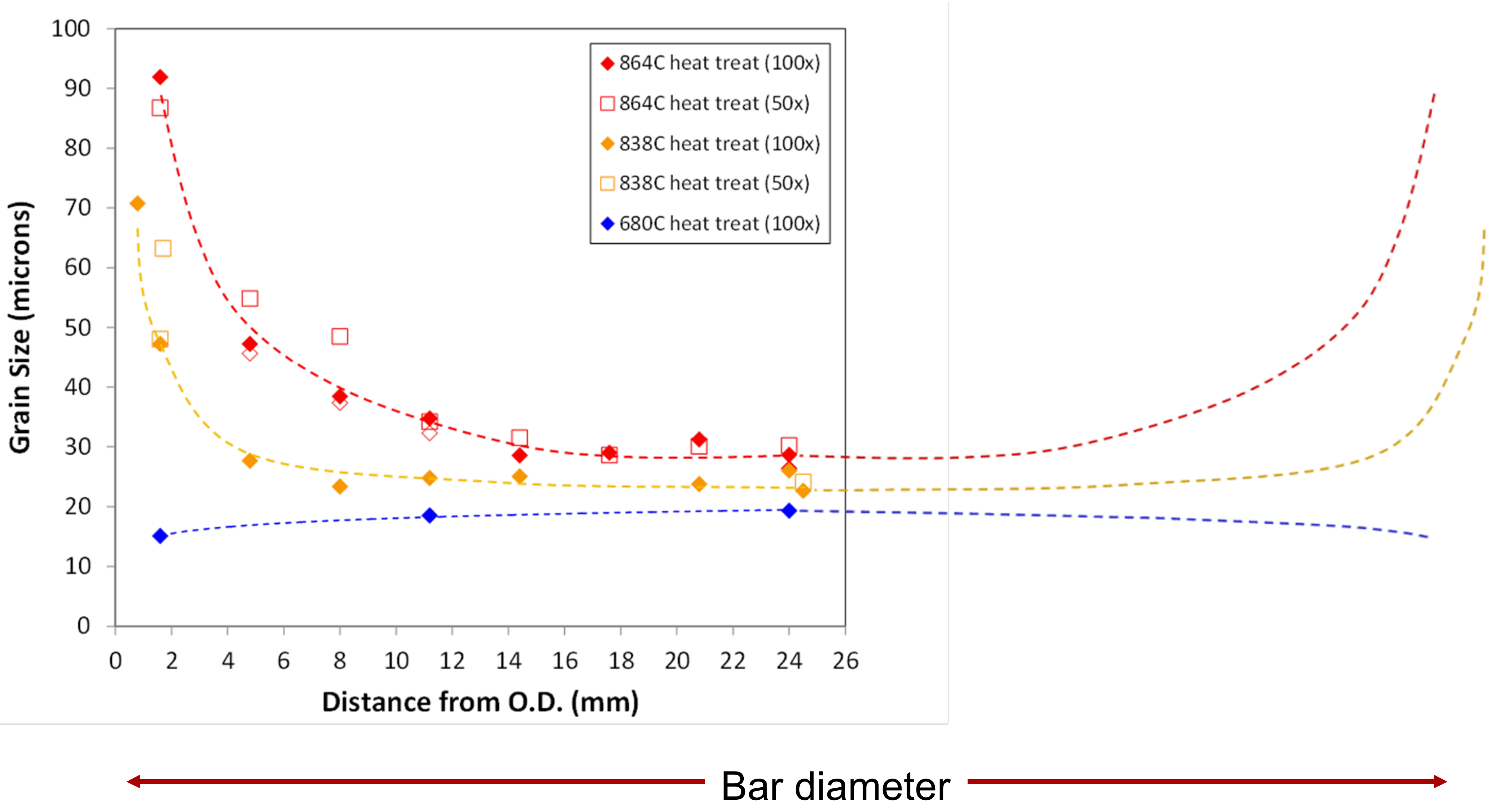






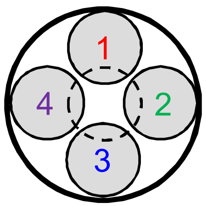
EDM machined tensile bar

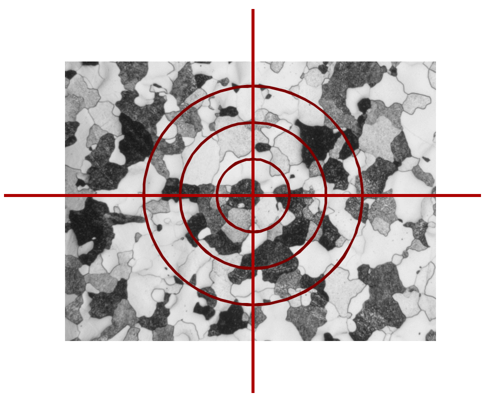
Each data point is avg. and std. deviation of 4 or 5 grain size measurements on individual micrographs

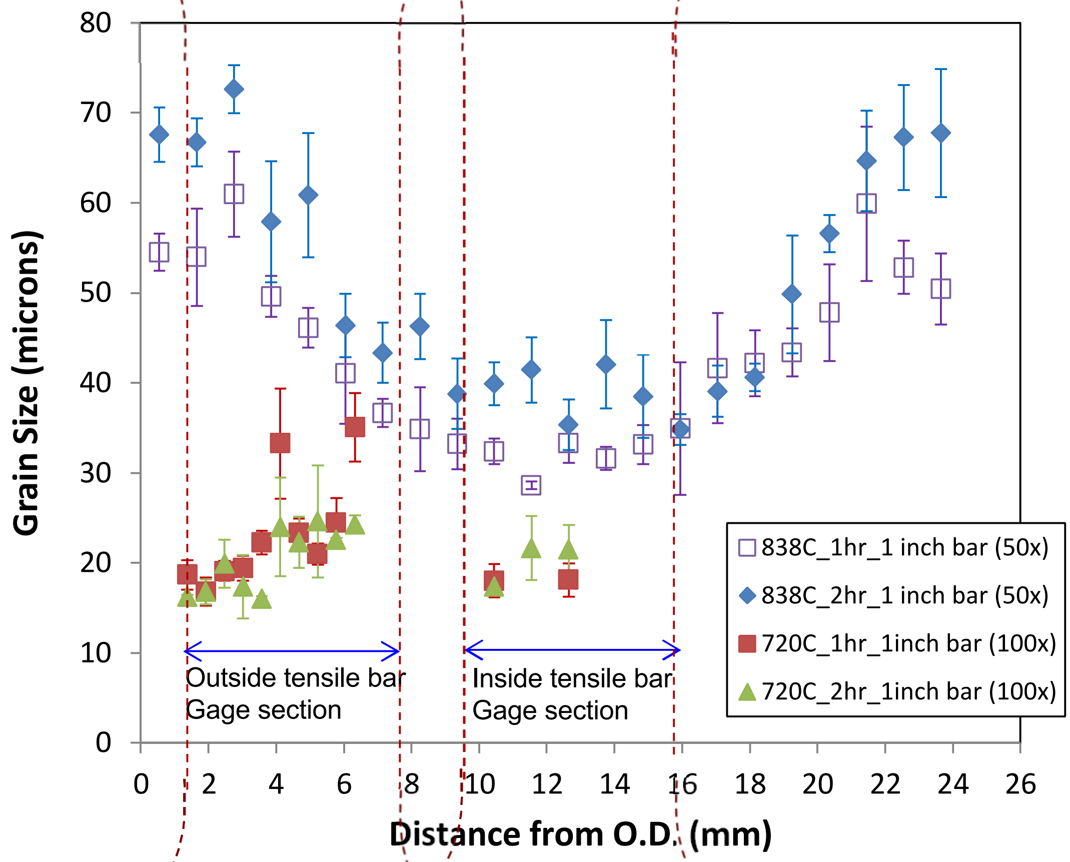


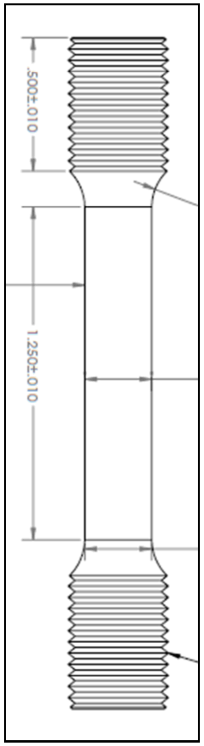
864C heat treat (100x)
864C heat treat (50x)
838C heat treat (100x)
838C heat treat (50x)
680C heat treat (100x)
Grain Size (microns)
100
90
80
70
60
50
40
30
20
10
0
0
2
4
6
8
10
12
14
16
18
20
22
24
26
Distance from O.D. (mm)
Bar diameter

Grain Size of 1-inch Diam. Bar





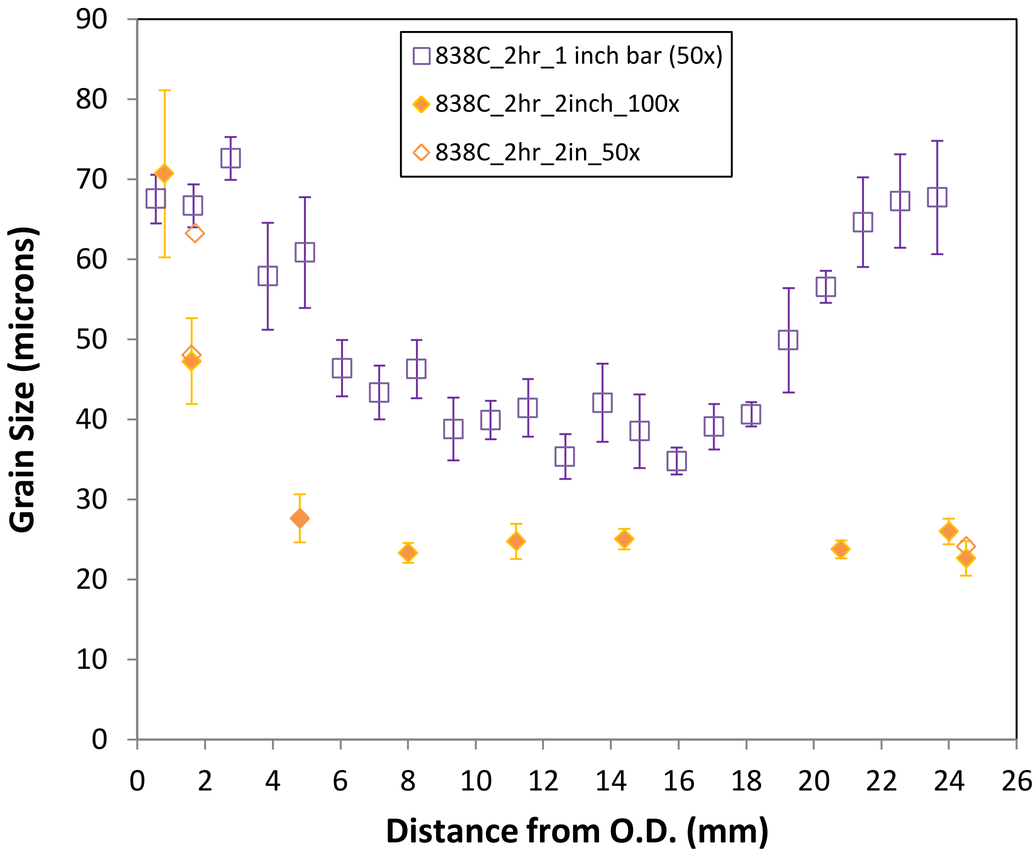




Each data point is avg. and std. deviation of 4 grain size measurements on individual micrographs

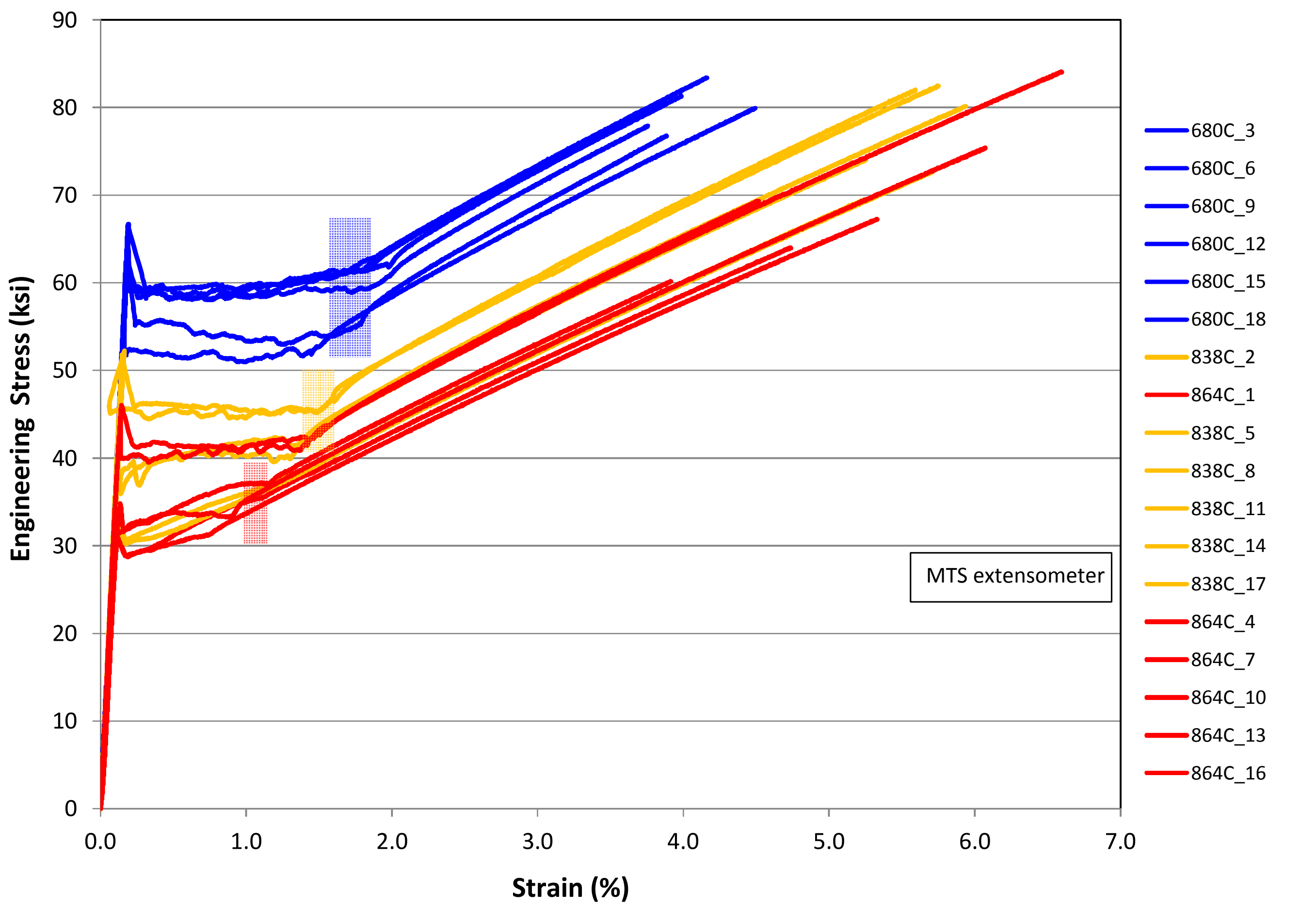
Grain Size Comparison: 1 in. Diam. vs. 2 in. Diam. Bar

- Grain size is actually slightly large in 1inch diameter bar for identical heat treatment (2hrs, 838C)
- Except near the outside edge of the bar



Examples of Tensile Test Response: New 2 in. Bar

- Most samples, not all, display upper/lower yield point behavior
- 0.2% offset yield strength (lower yield point) chosen for correlation to grain size data
- Extent of discontinuous yielding varies with heat treatment
- Ductility generally LOW



Hall-Petch Relationship

- Grain boundaries are an impediment to deformation (slip)
- Small-grain material has higher strength than coarse-grain material (assuming all other strengthening mechanisms are equal)
- Expressed in the Hall-Petch power law relationship:

$$\sigma_y = \sigma_o + kd^{-1/2}$$

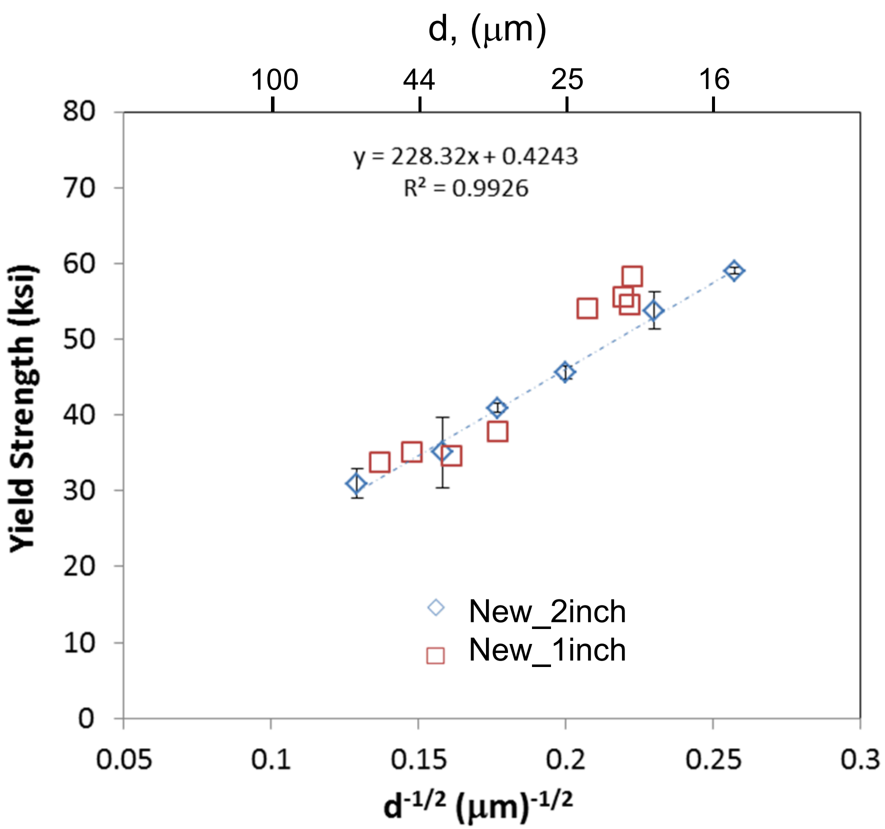
σ_y: yield strength

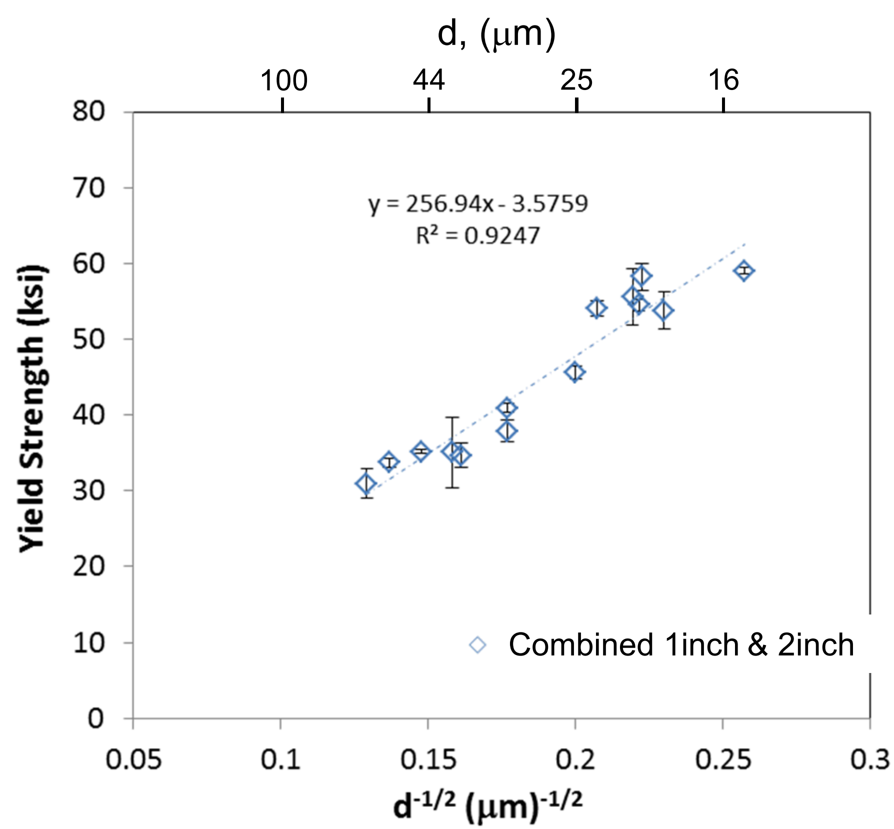
σ_o: intrinsic (friction) stress

k: constant (Hall-Petch coefficient)

d: grain size

Results: New Lots of Hiperco® (1in. and 2 in.)

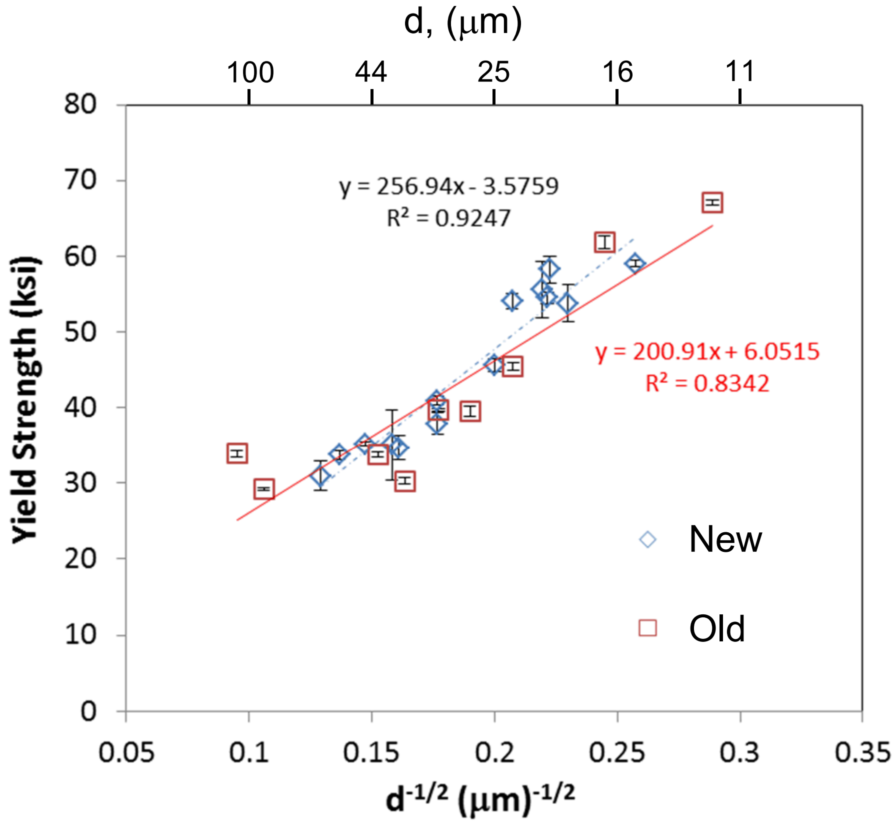


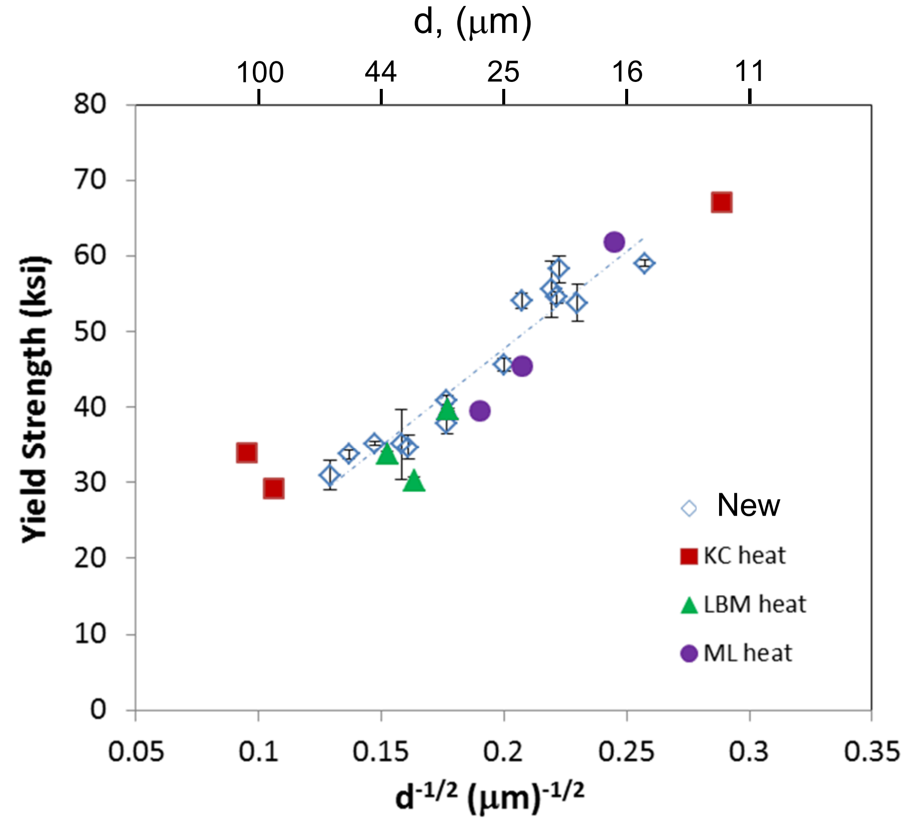


Data includes:
4 heat treat temperatures
2 heat treat times

Bar diam (inches)	Heat treatment and location	grain size microns
2	864C, outside	60
2	864, inside_new	32
2	838, outside_new	40
2	838, inside_new	25
2	680, outside_new	15.1
2	680, inside_new	18.9
1	838C_1hr, outside	46
1	838C_1hr, center_new	32
1	838C_2hr, outside_new	53.5
1	838C_2hr, center_new	38.5
1	720C_1hr_center_new	20.8
1	720C_2hr_center_new	20.2
1	720C_1hr_outside_new	23.3
1	720C_2hr_outside_new	20.4

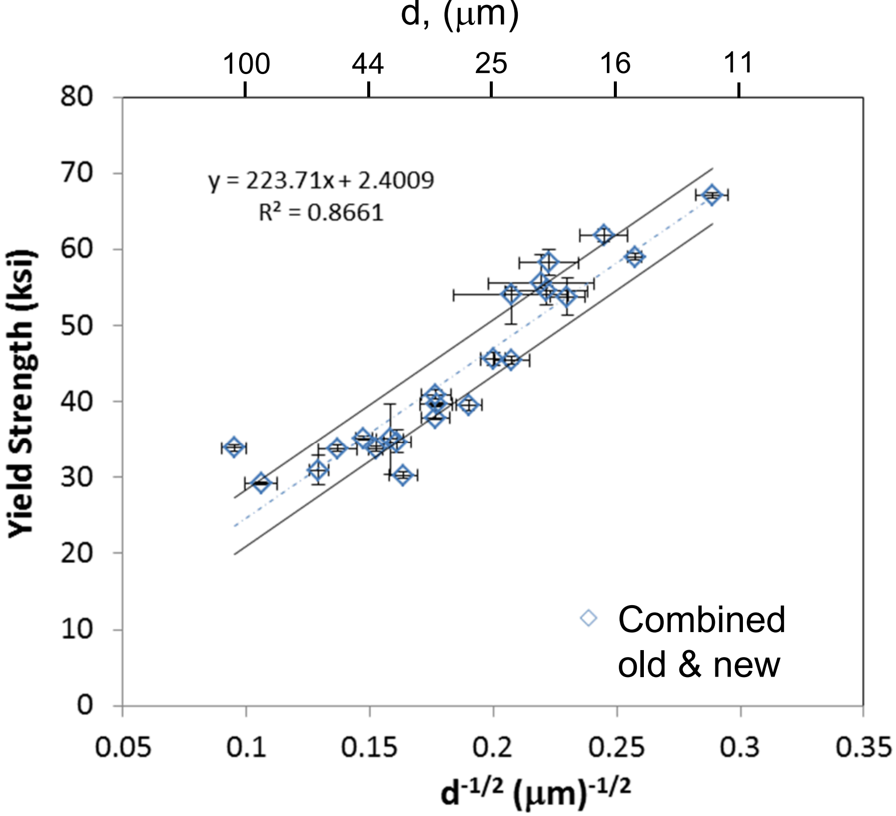
Compare to Historic Bar (2008) 1 in. and 1 3/8" diam.

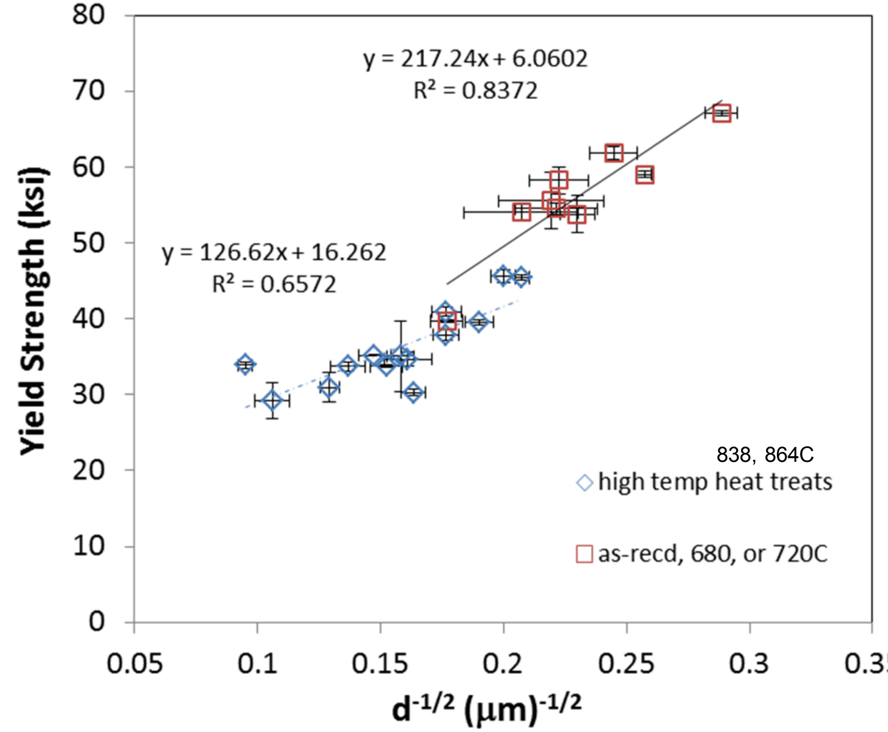




- Note: New tensile data avg. of ~6 tests for each data pt.,
Old lots avg. of ~3 tensile tests per data pt.
- New metallography, grain size measurement on bar slices
Old metallography, grain size measurement on grip ends of tensile bars

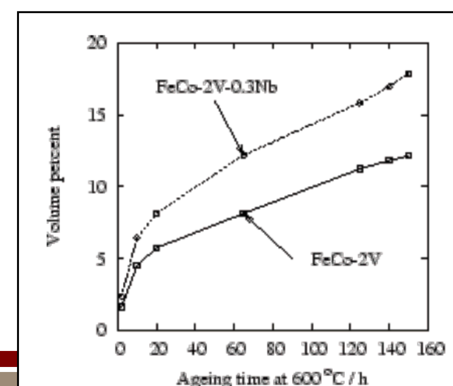
Precipitation during Low Temperature Heat Treatments?





- Horizontal and vertical error bars
- Statistical analysis applied: +/- one std. deviation of y-intercept are shown
- Recent heat treatment at 754°C not yet analyzed…
- Precipitation can alter the Hall-Petch slope, Precipitation shown previously by some authors →

Precipitates at lower heat treat temps. Yu et al., 2000



Results In Context

Literature on Fe-Co, Fe-Co-V alloys

Cold Rolled Sheet

Stoloff and Davies, 1964

Davies and Stoloff, 1966

Jordan and Stoloff, 1969

Thornburg, 1969

Moine et al., 1971

Branson et al., 1980

Thomas, 1980

Kawahara, 1983 (I and II)

Pitt and Rawlings, 1983 (forged 20%?)

Major and Orrock, 1988

Duckham et al., 1993

Fingers and Kozlowski, 1997

Fingers, 1999

Shang et al., 2000

Yu et al., 2000

Ren et al., 2001

George et al., 2002

Sourmail review (2008?)

Nabi et al., 2014

Bar

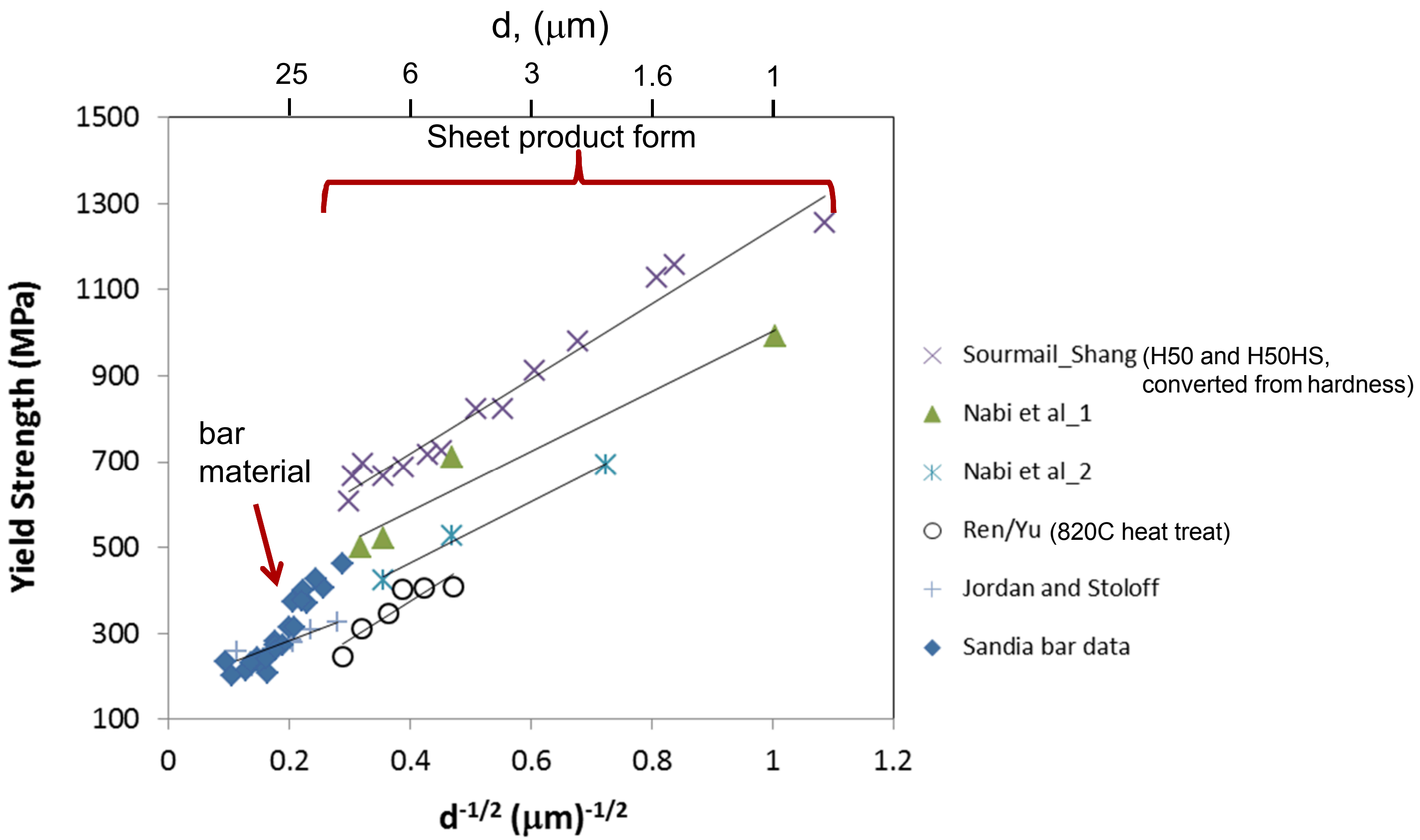
Fong et al., 1973

Zhao and Baker, 1994 (50Fe/50Co)

Sandia, 2014

References in red contain both grain size and yield strength data

Comparison to Previous Studies (Cold Rolled Sheet)



- Bar results: comparable or stronger than sheet of similar grain size
- Could other methods to reduce grain size in bar produce comparable strength to "typical" sheet material?
- Effects on ductility, magnetic properties are unknown

One Previous Study on Barstock (50/50 FeCo)

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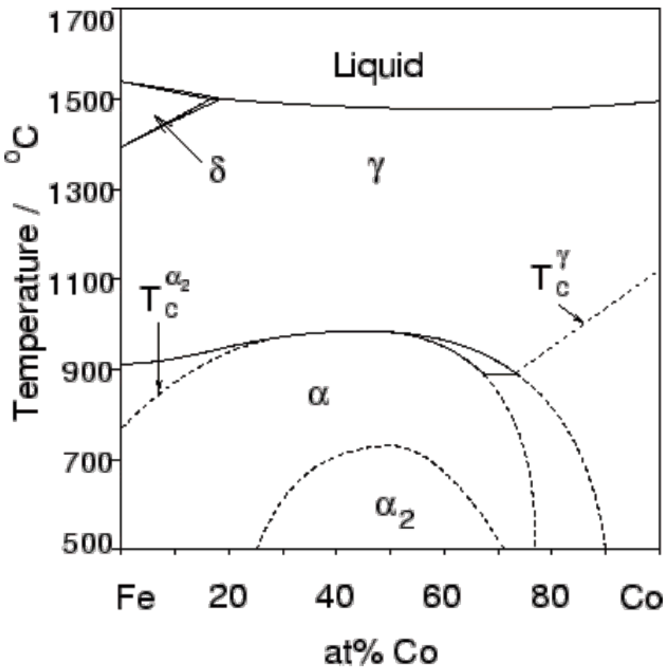


Figure: The Fe-Co binary diagram as given in [5]. T_c denotes the Curie temperature.

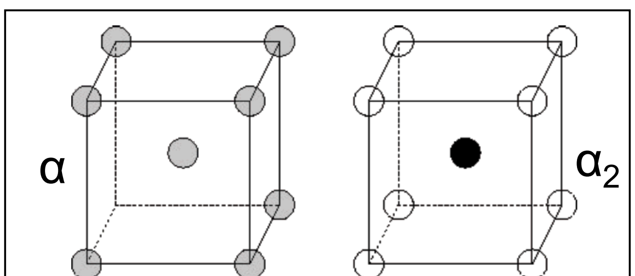
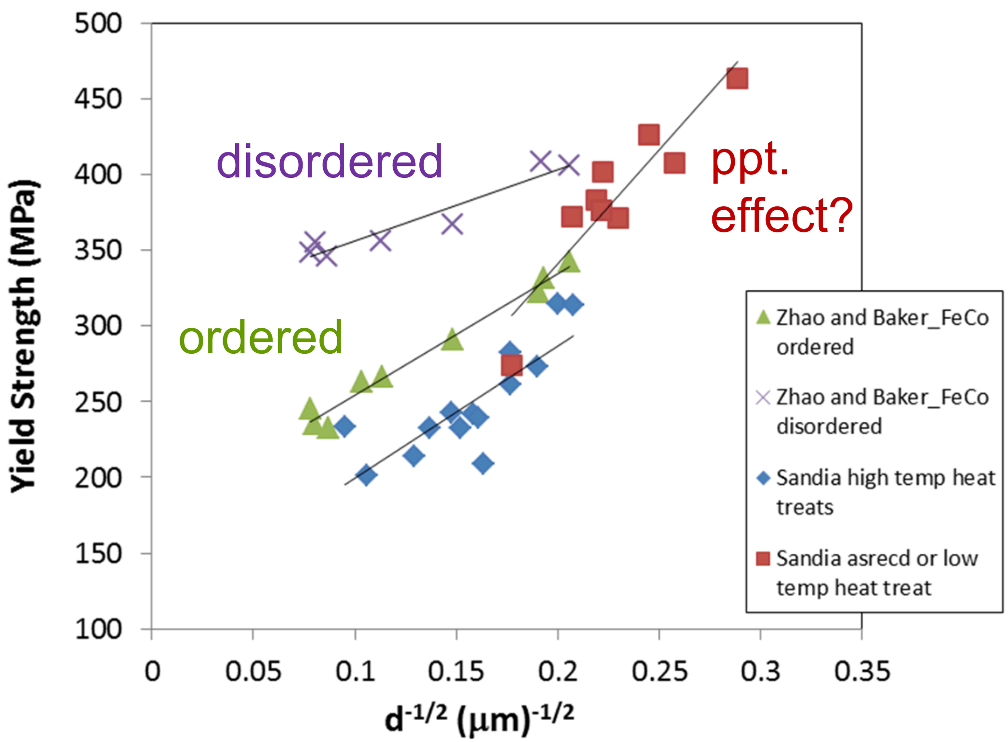
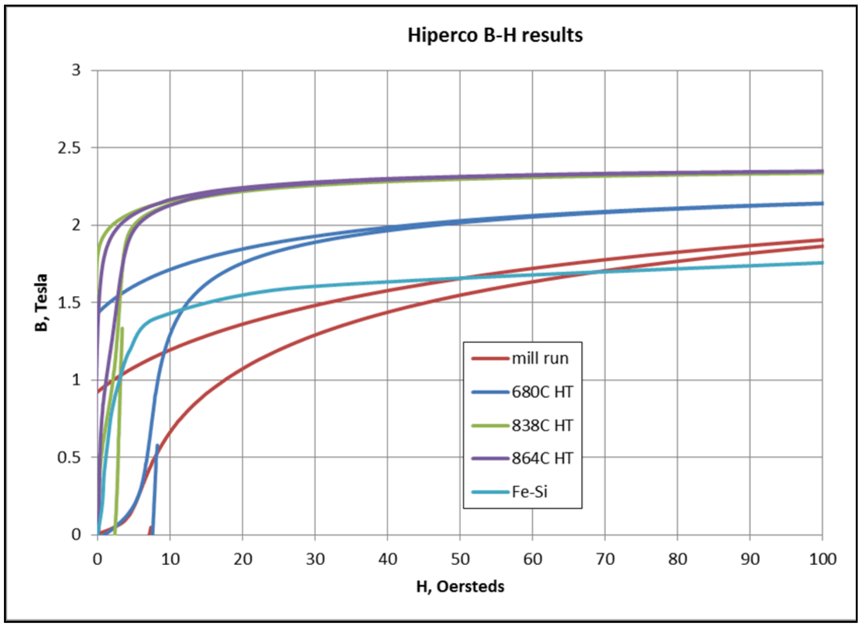


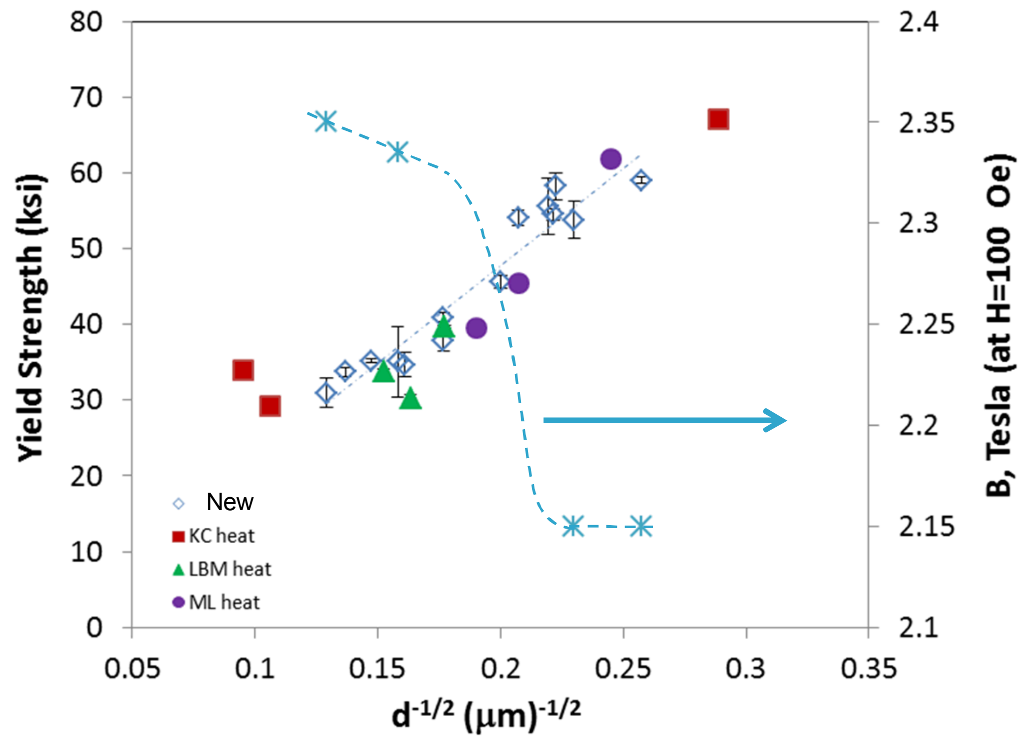
Figure: b.c.c. and ordered B2 structure.



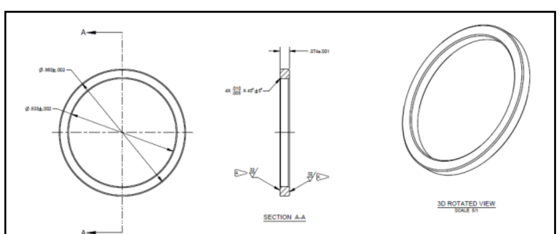
L. Zhao and I. Baker, *Acta Met. Mat.*,1994.

Tradeoff with Magnetic Performance





B-H test ring



Summary

- Grain size gradient across bar diameter -- larger grains near outside of bar. Near mid-radius, a duplex grain structure is formed with remnants of the forging deformation.
- Duplex grain structure produces variability (larger error bars) in grain size measurements, but for each test condition, a single average grain size is needed for Hall-Petch correlation.
- **Nevertheless, yield strength of Hiperco® bar correlates well to grain size through the Hall-Petch relationship.**
- Historical data matches fairly well with recent bar lots with 500 ppm Nb.
- The data significantly enriches the low strength/large grain size region of the overall Hall-Petch plot for Hiperco® (and related alloys).

- **Processing methods to reduce grain size below 10 microns could increase strength in bar to comparable levels reported for sheet. Could fine grain size be beneficial to ductility as well?...while maintaining good magnetic response?**

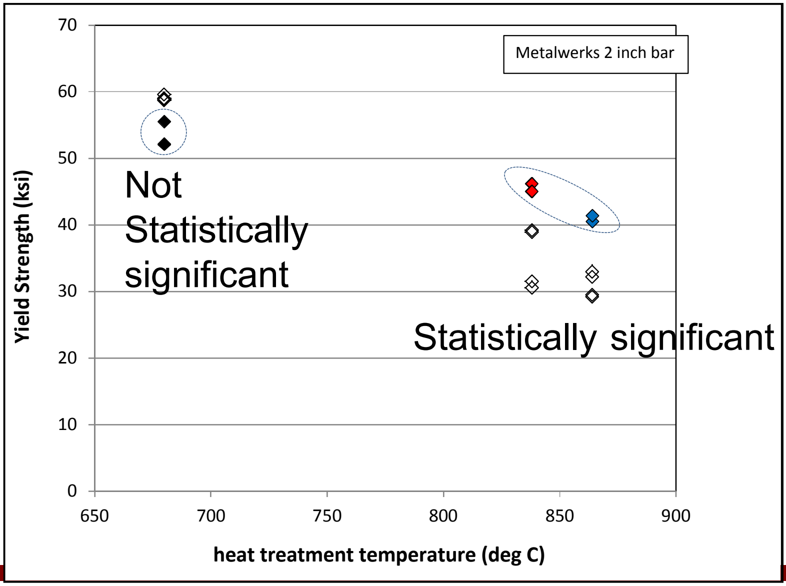
Thanks to Dr. Lisa Deibler for additional grain size measurements and to Clinton Holtey (Sandia) and KJS Associates, Inc. for magnetic measurements.

QUESTIONS?

BACKUP SLIDES

Statistical significance of “inside” vs. outside samples

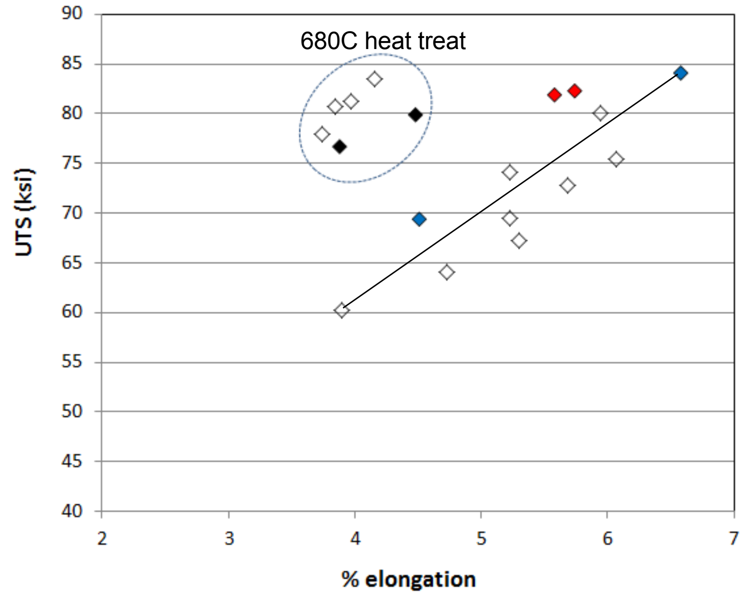
680C heat treat t-test		
t-Test: Two-Sample Assuming Equal Variances		
	Variable 1	*Variable 2*
Mean	59.05	53.8
Variance	0.15	5.78
Observations	4	2
Pooled Variance	1.5575	
Hypothesized Mean Difference	0	
df	4	
t Stat	4.857521	
P(T<=t) one-tail	0.004147	
t Critical one-tail	2.131847	
P(T<=t) two-tail	0.008294	
t Critical two-tail	2.776445	



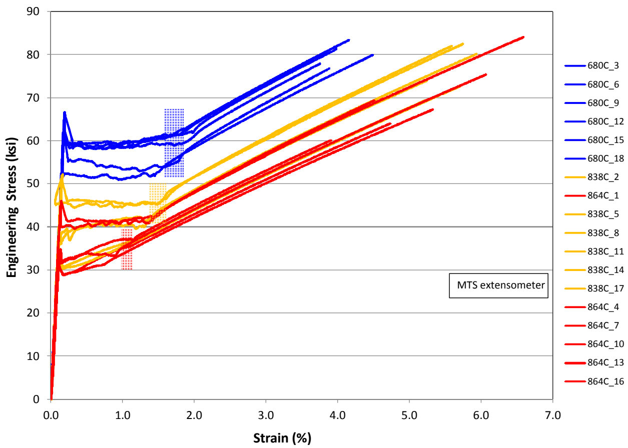
838C heat treatment t-test		
t-Test: Two-Sample Assuming Equal Variances		
	Variable 1	*Variable 2*
Mean	35.05	45.6
Variance	21.48333	0.72
Observations	4	2
Pooled Variance	16.2925	
Hypothesized Mean Difference	0	
df	4	
t Stat	-3.01806	
P(T<=t) one-tail	0.019619	
t Critical one-tail	2.131847	
P(T<=t) two-tail	0.039238	
t Critical two-tail	2.776445	

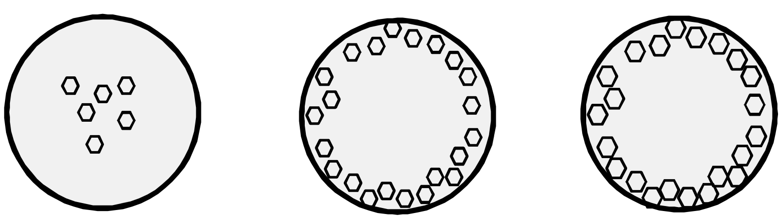
864 C heat treat t-test		
t-Test: Two-Sample Assuming Equal Variances		
	Variable 1	*Variable 2*
Mean	30.975	40.95
Variance	3.6425	0.405
Observations	4	2
Pooled Variance	2.833125	
Hypothesized Mean Difference	0	
df	4	
t Stat	-6.84305	
P(T<=t) one-tail	0.001193	
t Critical one-tail	2.131847	
P(T<=t) two-tail	0.002386	
t Critical two-tail	2.776445	

- The material does not "neck". Higher strength directly correlates to higher ductility, but all ductility values are LOW.

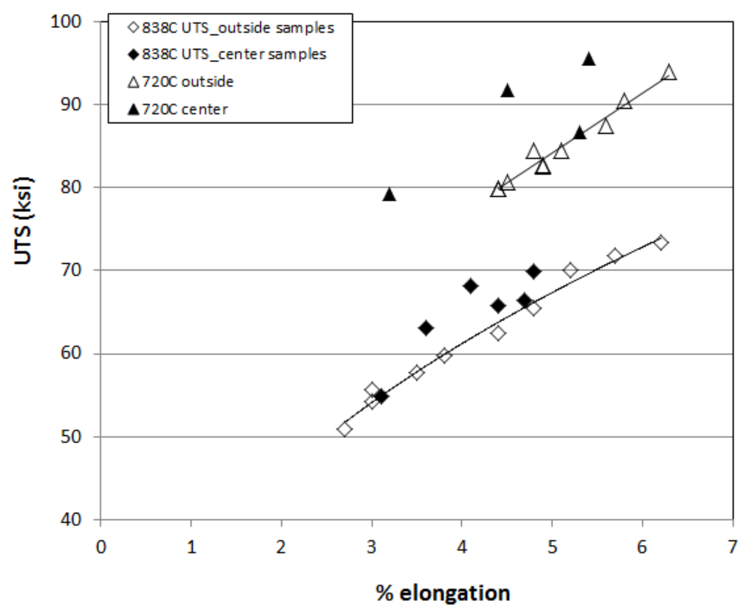


2 inch bar





680C 838C 864C



1 inch bar