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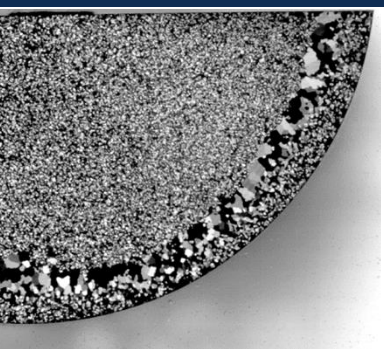

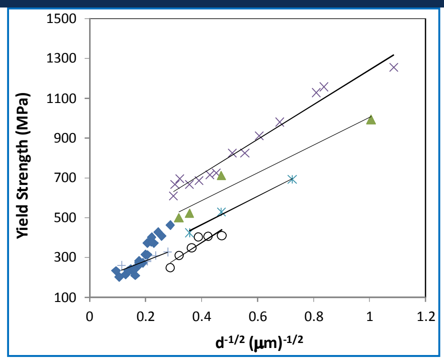

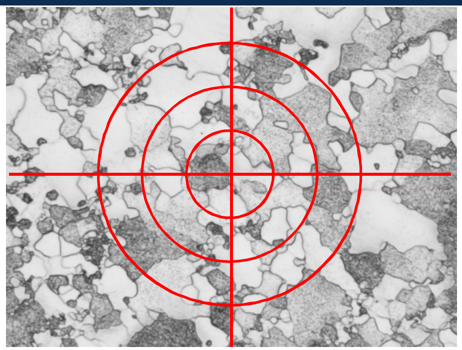

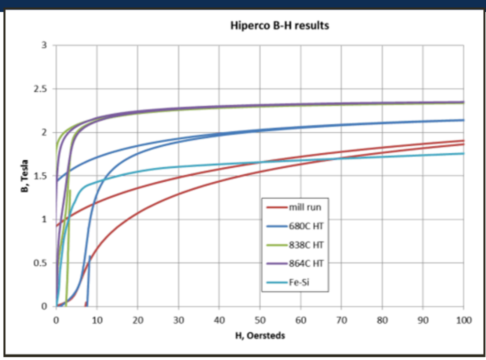

ECAE of Hiperco Soft Magnetic Alloy

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Dr. Ibrahim Karaman, Taymaz Jozaghi
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October 17, 2016

U.S. DEPARTMENT OF ENERGY
National Nuclear Security Administration
LDRD LABORATORY DIRECTED RESEARCH & DEVELOPMENT

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Collaborators

Jeff Rodelas and Mike Maguire (Sandia) – Inertia Welding

Dr. Ibrahim Karaman and Taymaz Jozaghi (Texas A&M) – Equal Channel Angular Extrusion (ECAE)

Rick Kellogg, Sara Szarka, Patrick White (Sandia)

Topics

Introduction and History

Equal Channel Angular Extrusion (ECAE)/Pressing (ECAP)

Microstructure of ECAE Material, EBSD/Texture

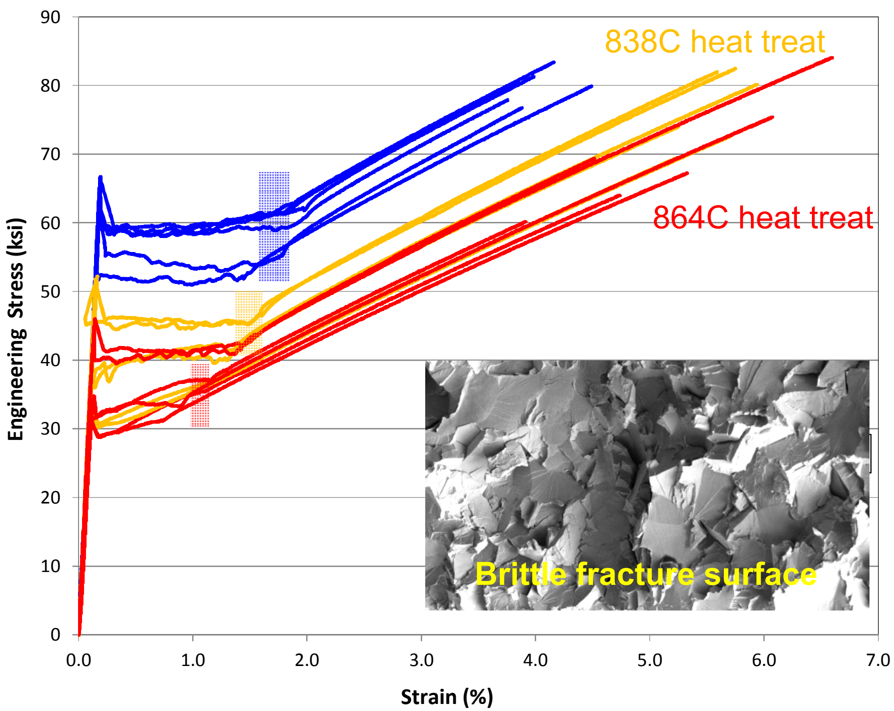
Tensile Properties

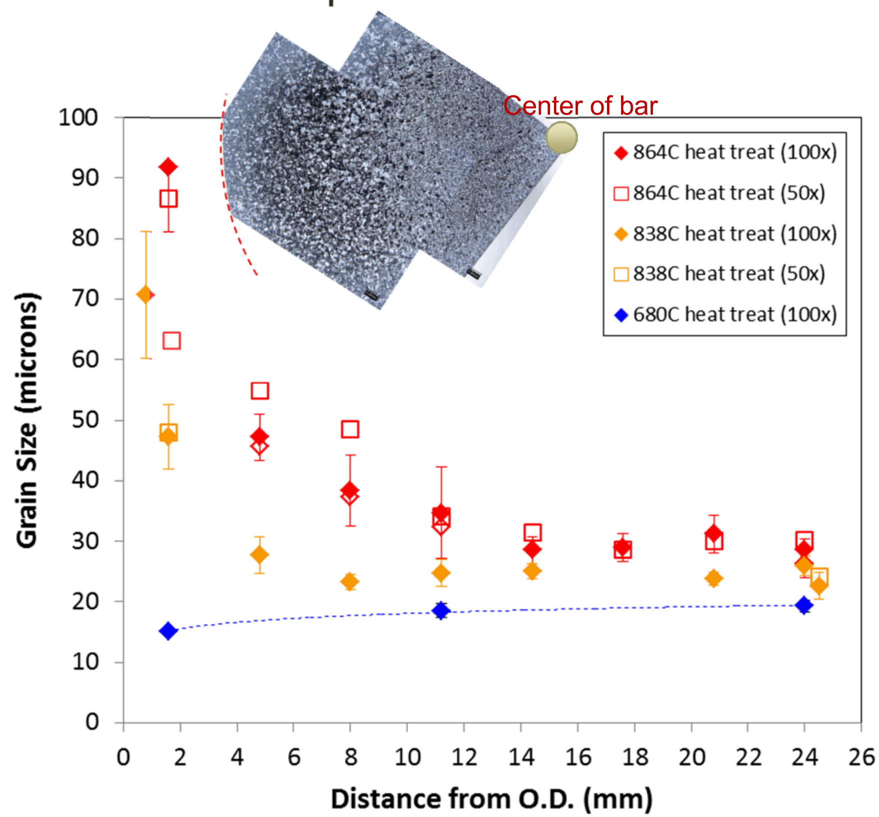
Magnetic Properties, EBSD/Texture

Summary

Introduction: Hiperco 50A Soft Magnetic Alloy

- Hiperco (Fe-Co-2V) undergoes **brittle** fracture, with low strength in bar form
- The microstructure and properties of bar are also **non-uniform**
- BUT, it has the highest magnetic saturation of any commercial alloy
- Used in solenoids, electric motors, and other components





Why does Hiperco have poor mechanical properties?

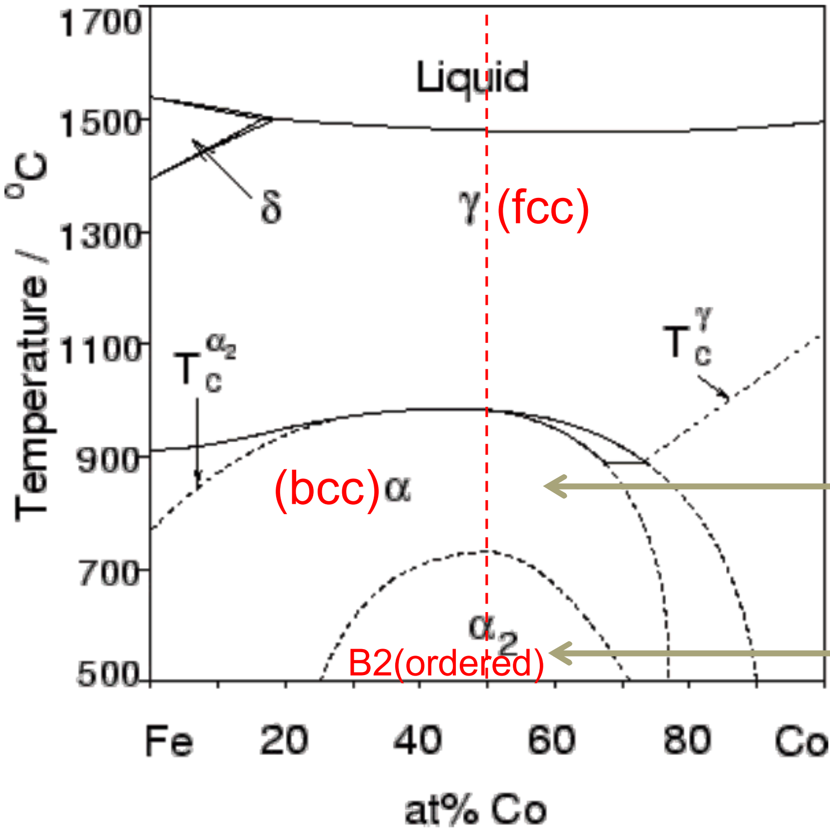
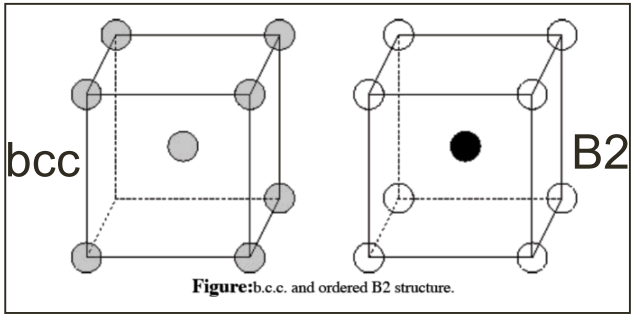


Figure: The Fe-Co binary diagram as given in [5]. T_c denotes the Curie temperature.



Crystal structures of Hiperco alloy

bcc: Good mechanical properties, So-so magnetic properties

B2: Ordered structure has poor mechanical properties, Excellent magnetic properties

- Disordered bcc material is generally ductile. Conventional bar is low-strength.
- Ordered FeCo is brittle and low strength. Addition of 2%V provides some ductility, allowing conventional forming processes and heat treatment

Previous Research

Sandia National Laboratories

- Zhao and Baker (1994) (Conventional) "Extrusion Processing of FeCo", *J. Materials Science*
  - 750C, 1000C extrusion temperatures
  - Fe-30Co, Fe-50Co, Fe-70Co
  - 20-100 μm grain size range
- Kawahara et al. (1980s)*:* studied the effects of cold-work on ***sheet*** material
  - High strength ***and higher ductility*** are possible with high amounts of cold-work >=~80%
  - Attributed to local concentration disordered (LCD) zones
- Many researchers (1960s-2000s)
  - Hall-Petch behavior in sheet and bar FeCo-V material
  - Various researchers attribute improved ductility to 2nd phase particles, very fine grain size, partial recrystallization with subgrains to prevent dislocation pileups and crack propagation (slide partitioning/slip dispersion) (George et al, 2002, Stoloff et al, 1964-1970, Pitt and Rawlings, 1983, Thornburg, 1969)

Two Current Research Areas

1. **Inertia Welding: Solid-state weld Hiperco to an alloy with better mechanical properties. Locate the Hiperco where good magnetic properties are needed and the other alloy where good mechanical performance is needed.**

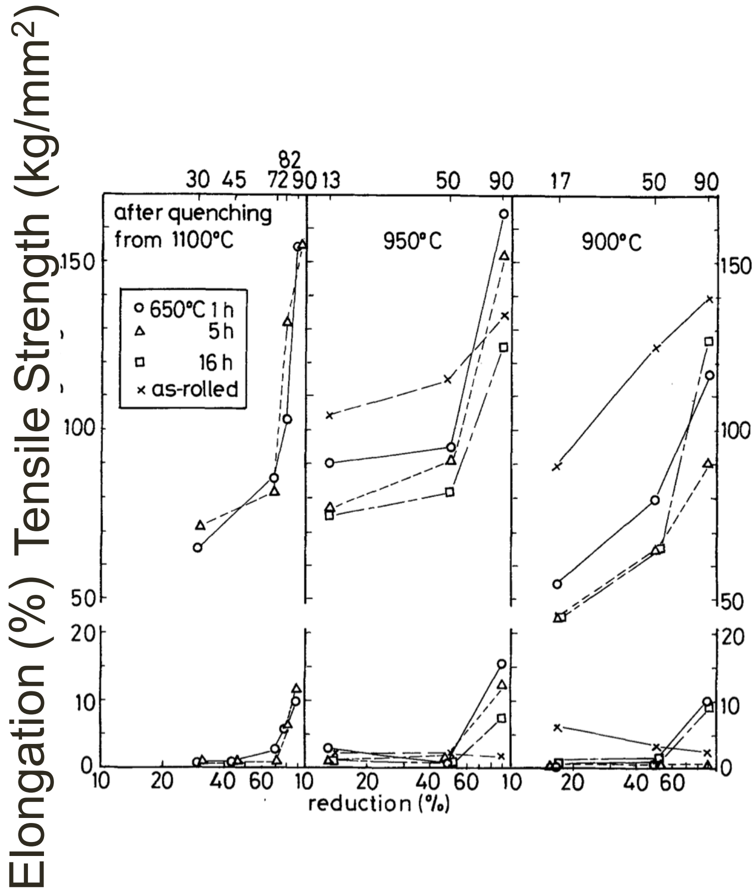
2. **Equal Channel Angular Extrusion/Pressing (ECAE/ECAP): Improve the mechanical properties of Hiperco. Try not to sacrifice too much magnetic performance.**

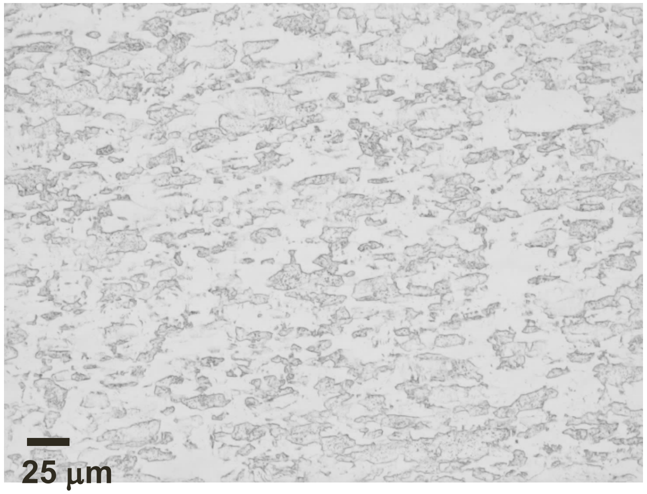
Comparison to Sheet

Forged bar Hiperco is weak, brittle, and inhomogeneous. In contrast, *sheet rolled* Hiperco is stronger and can be more ductile, with adequate magnetic performance.

High levels of cold work (prior to ordering) enhances strength & ductility

Kawahara K., J. Mat. Sci. 18:3437-48 (1983)





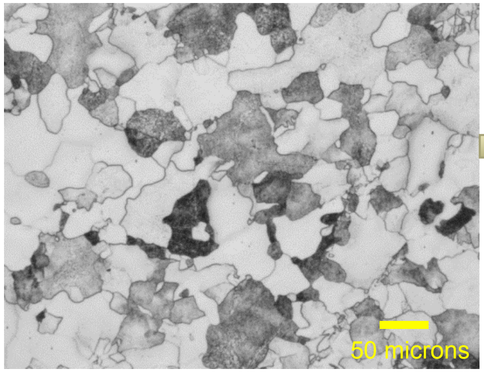
Fine grain microstructure of sheet

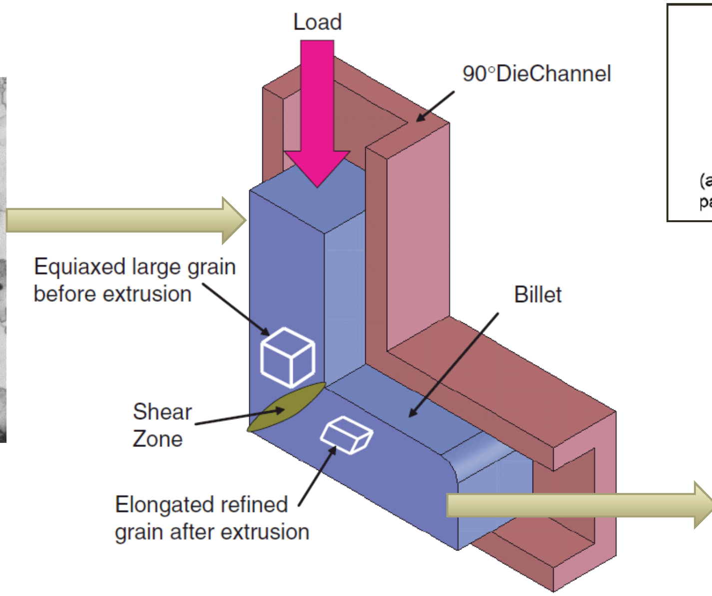
Current Research: Equal Channel Angular Extrusion (ECAE), a.k.a. Equal Channel Angular Pressing (ECAP)

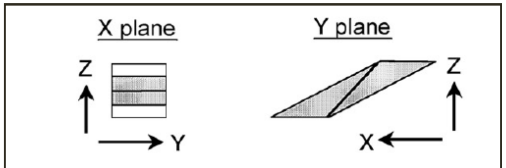
The premise: Sandia components would benefit greatly from increased mechanical strength and/or ductility in Hiperco.

The challenge: Is it possible to produce sheet-like mechanical properties in bar? And can the magnetic performance be maintained?

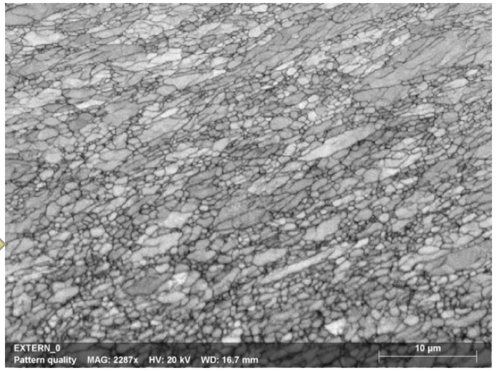
Equal Channel Angular Pressing (ECAP) produces severe plastic deformation ***without a change in cross-section***; ***nanostructured material* with increased strength**.



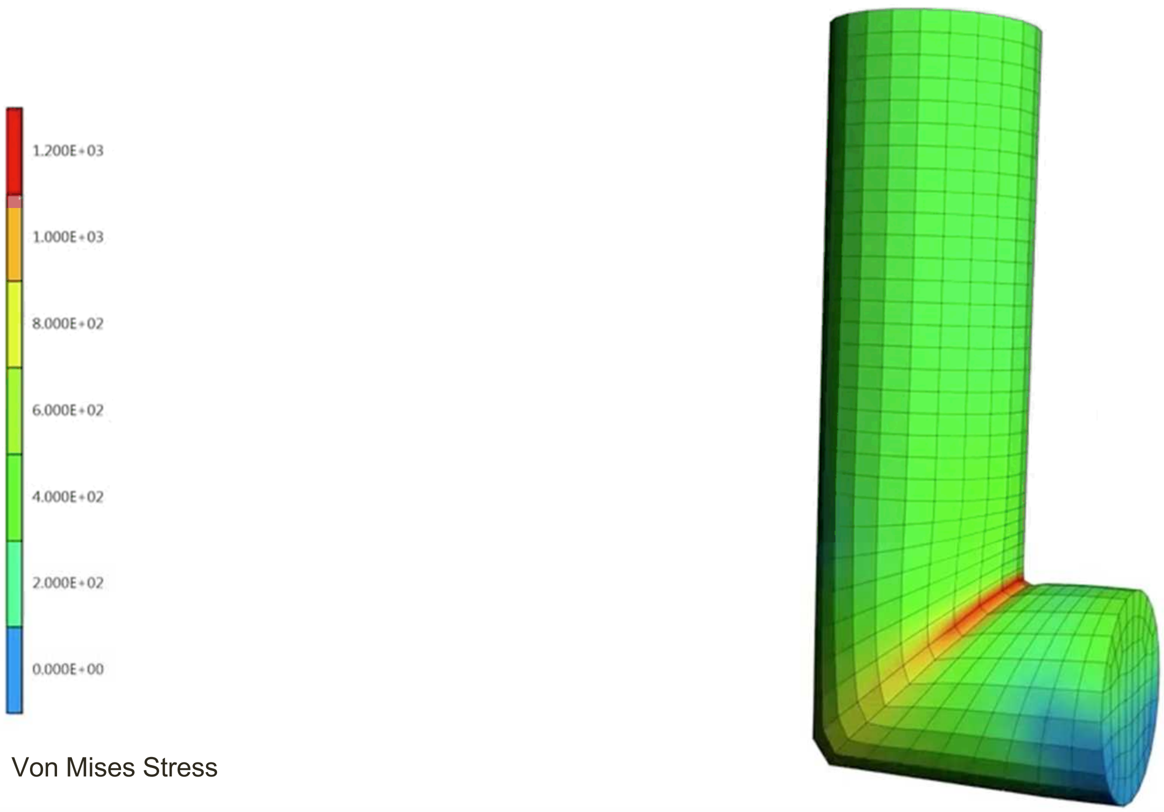




(a) The deformation of a cubic element on a single pass [33].

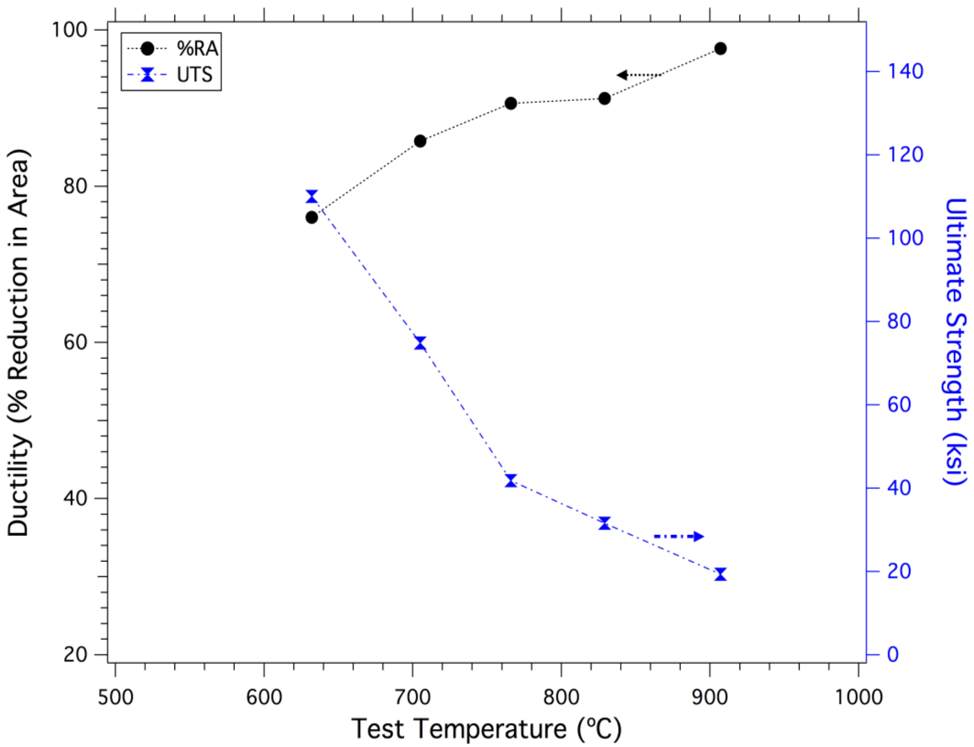


Equal Channel Angular Extrusion/Pressing



A.V. Shutov and R. Kreißig, *Comp. Methods in Applied Mech. and Eng.*, 197(21-24), pp 2015-29, 2008.
A.V. Shutov et al., *Materialwissenschaft und Werkstofftechnik*, 43(7), pp 617-25, 2012.

High-Temperature Ductility of Hiperco

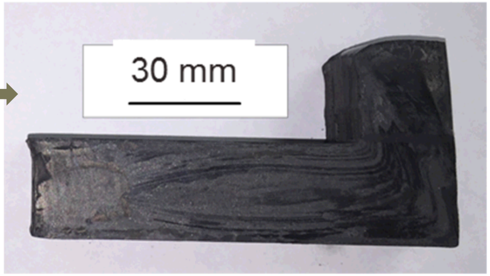


- High temperature tensile testing at Sandia shows Hiperco has appreciable ductility at high temperatures (≥~750˚C)
- Encouraging enough to proceed with ECAE trials *at high temperature*
- Collaboration with Texas A&M ECAE laboratory

11 ECAE trials completed at Texas A&M

HCO-1	4E 850C (WQ)	Microstructure, tensile, magnetic
HCO-2	1E 750C	Extrusion didn't finish
HCO-3	1E 850C / 2&3E 750C	Significant cracking and shear bands
HCO-4	1E 950C	Significant surface cracking
HCO-5	4E 850C / 5&6E 750C (WQ)	Microstructure, tensile
HCO-6	2C 850C / 3&4C 750C (WQ)	Microstructure, tensile
HCO-7	2C 850C / 3&4C 750C / 5C 700C	Significant shear bands and load increase, indicates transformation to B2 structure
HCO-8	2C 850C / 3&4C 750C (AC)	Same as HCO-6 except air cooled Tensile tests complete
HCO-9	1&2C-850C / 3&4C-750C (WQ)	Same as HCO6
HCO-10	1&2C-850C / 3&4C-750C (WQ)	Same as HCO6
HCO-11	1&2C-850C / 3&4C-750C (AC)	Same as HCO8

(All trials: 30 min heating prior to extrusion, Extrusion rate 0.5 in. per sec.)



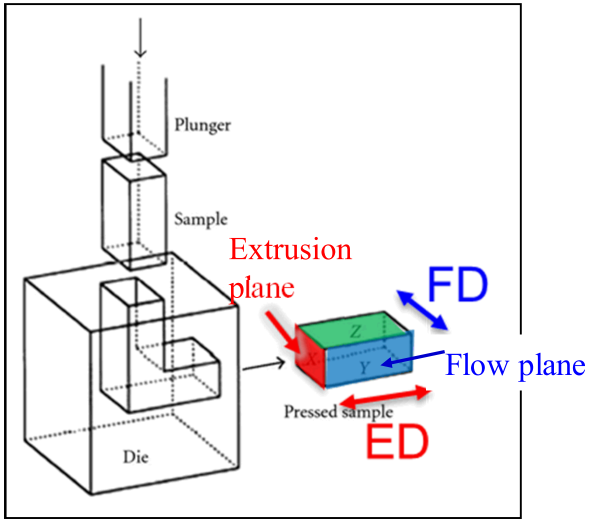
Results show ECAE at 850 → 750°C range can successfully produce material for testing

The sample is axially rotated between ECAE passes.

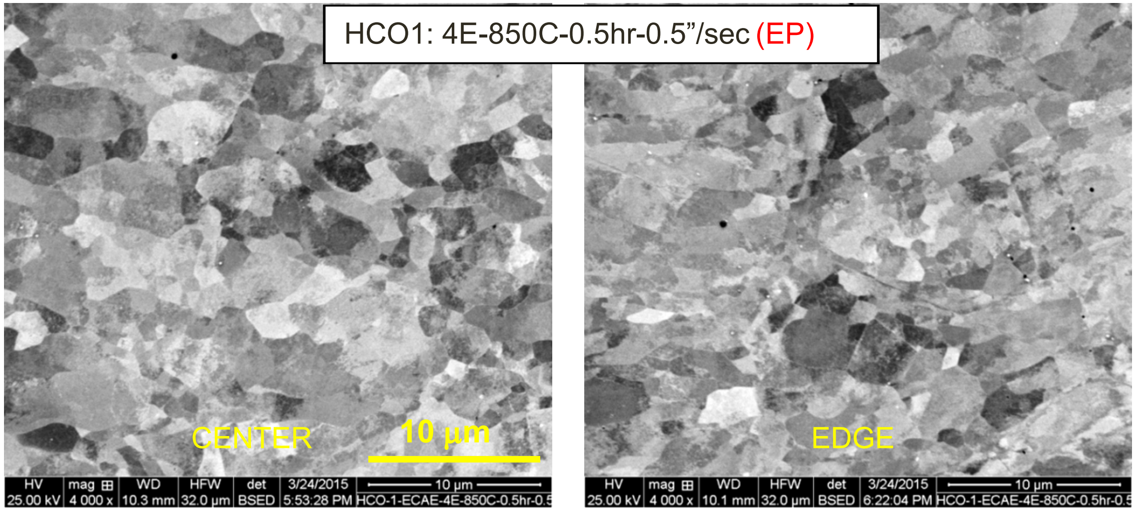
Route name	Min. # of passes	Billet rotations about the extrusion axis				Material Yield*	Effect on microstructure
		1 →	2 →	3 →	4 → N		
A	1	0°	0°	0°	etc.	0.58	elongation (lamellar)
B (B_A)	2	+90°	-90°	+90°	etc.	0.67	elongation (filamentary)
C	2	180°	180°	180°	etc.	0.83	back/forth shearing
C' (B_C)	4	+90°	+90°	+90°	etc.	0.67	back/forth cross-shearing
E	4	180°	90°	180°	etc.	0.78	back/forth cross-shearing

Scanning Electron Microscopy of ECAE Material

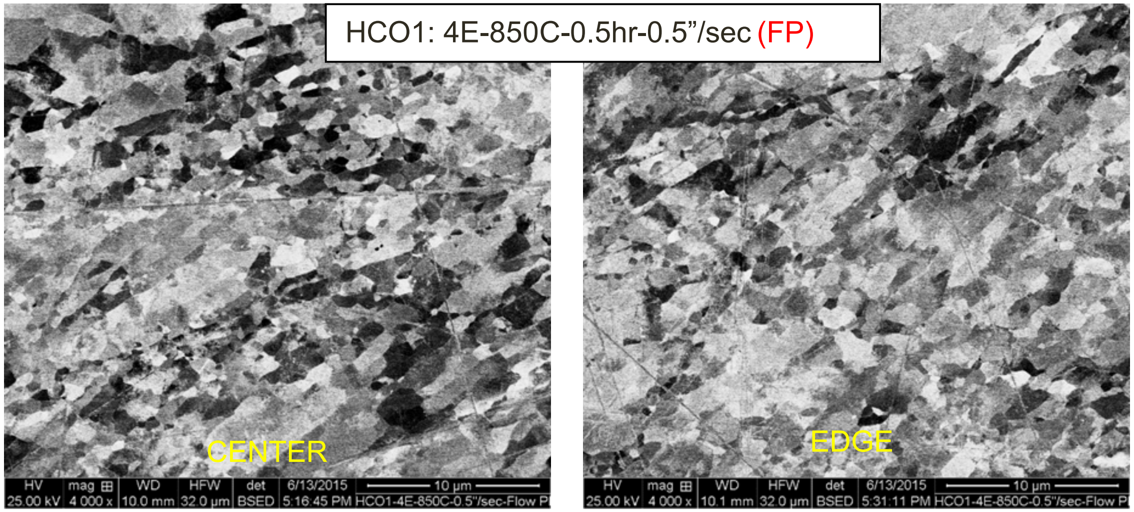
- Significant grain refinement achieved (2-3 micron grain size)
- Uniform microstructure from center to edge of billet
- Note, this Hiperco has 600 ppm Nb (meant to promote grain refinement)



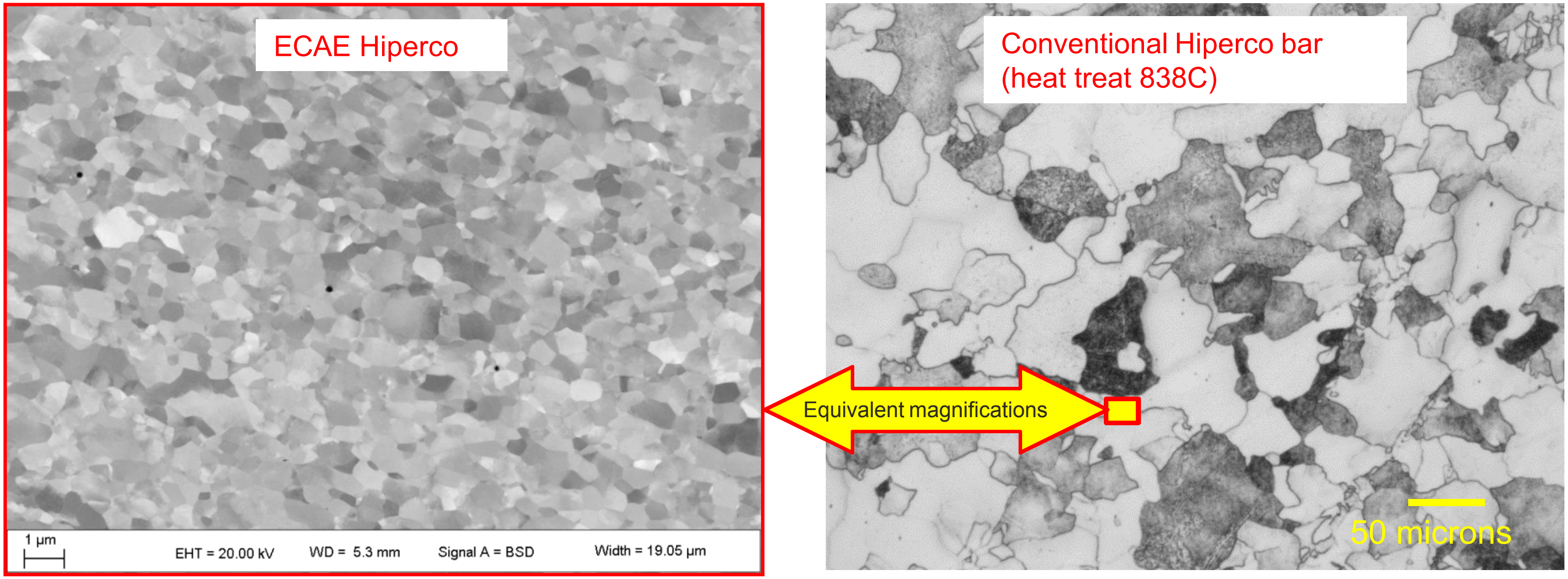
HCO1: 4E-850C-0.5hr-0.5"/sec (EP)



HCO1: 4E-850C-0.5hr-0.5"/sec (FP)



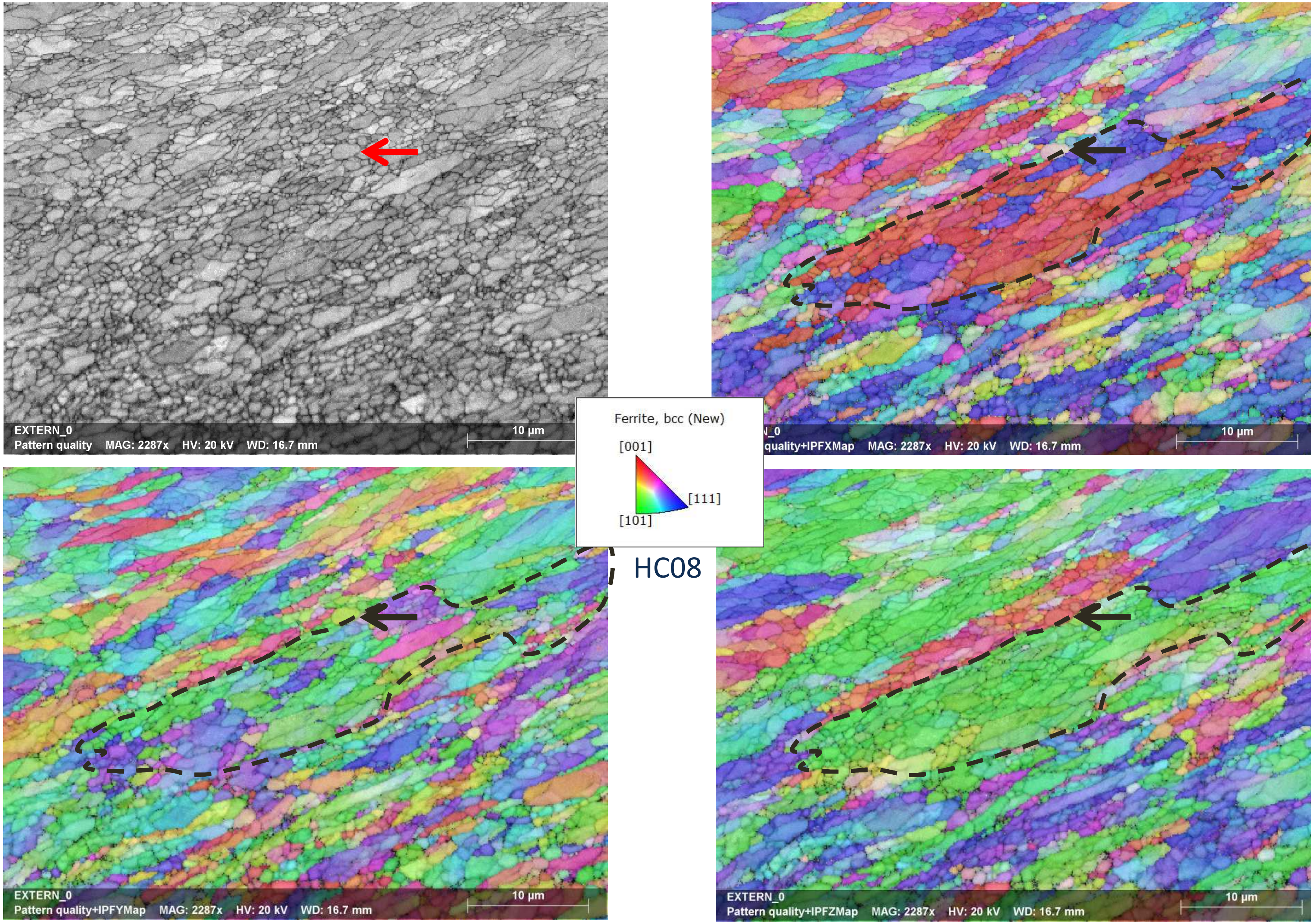
Microstructural Refinement Achieved



Comparison between as-ECAE material and conventional bar (heat treated)

- Entire ECAE image (same magnification) would fit inside the small red box
- ECAE approx. 1.5 micron grain size, conventional bar ~ 25-50 micron grain size

Current Work: EBSD/Texture Analysis



Texture in As-ECAE Condition

Literature (Beyerlein et al)

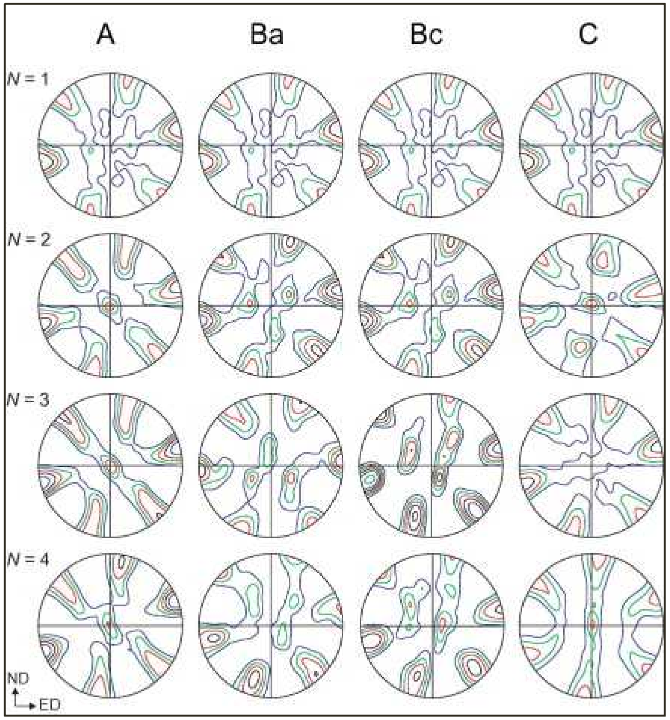
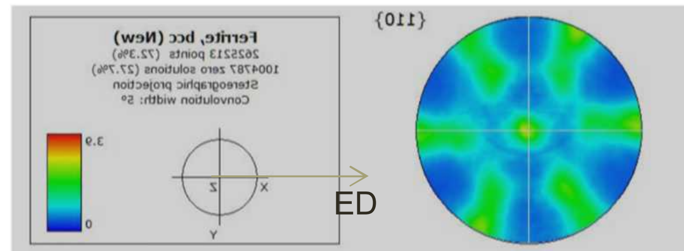


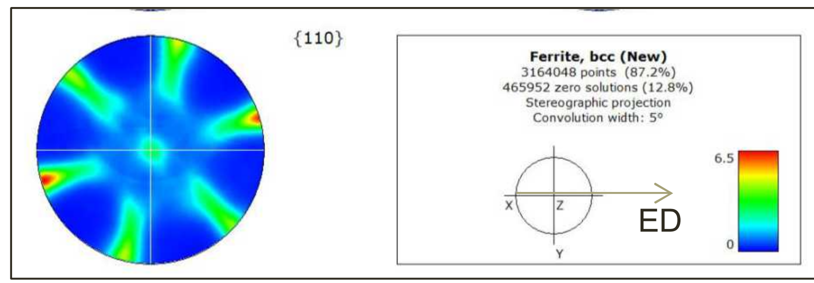
Fig. 9. (1 1 0) pole figures of experimental textures in IF-steel [32] after 1–4 passes (N) of ECAE via routes A, Ba, Bc and C. Isolevels: 1.0/1.3/1.6/2.0/2.5/3.2/4.0/5.0/6.4/8.0 mr.

(bcc material)

Irene J. Beyerlein, László S. Tóth **Texture evolution in equal-channel angular extrusion** *Progress in Materials Science*, Volume 54, Issue 4, 2009, 427–510
http://dx.doi.org/10.1016/j.pmatsci.2009.01.001

Hiperco HCO6 **Route C**
2 passes, 850C, 2 passes 750C
water quench





Hiperco HCO8 **Route C**
2 passes, 850C, 2 passes 750C
air cool

Texture in As-ECAE Condition

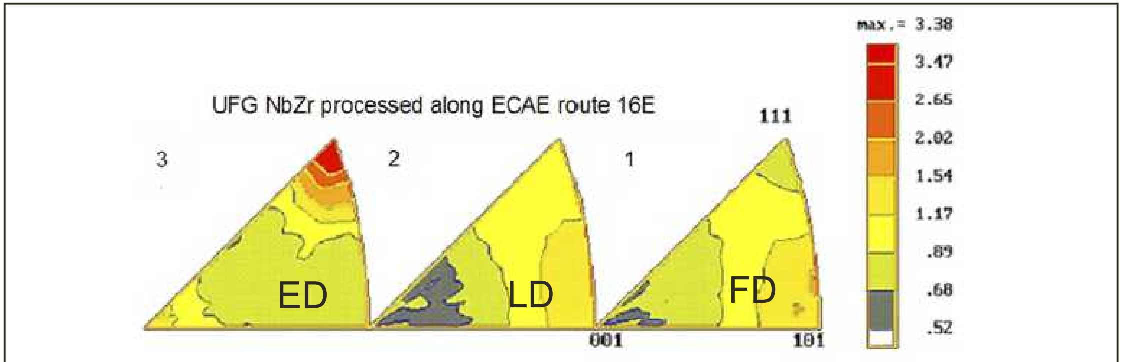
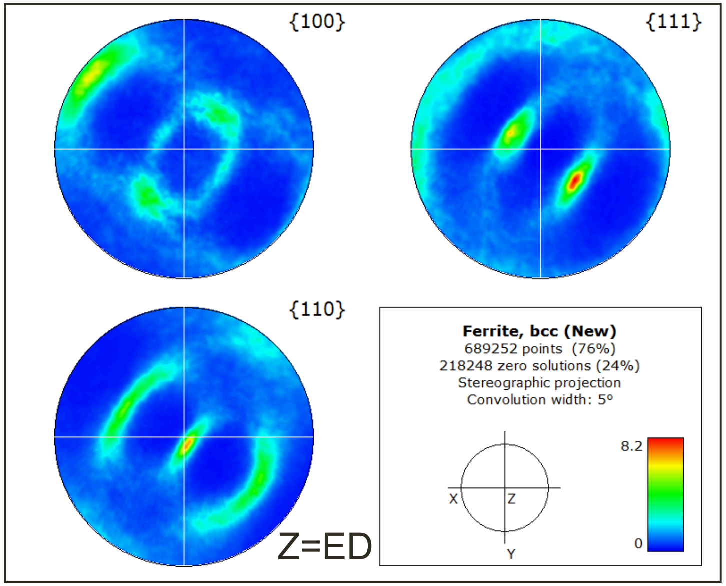
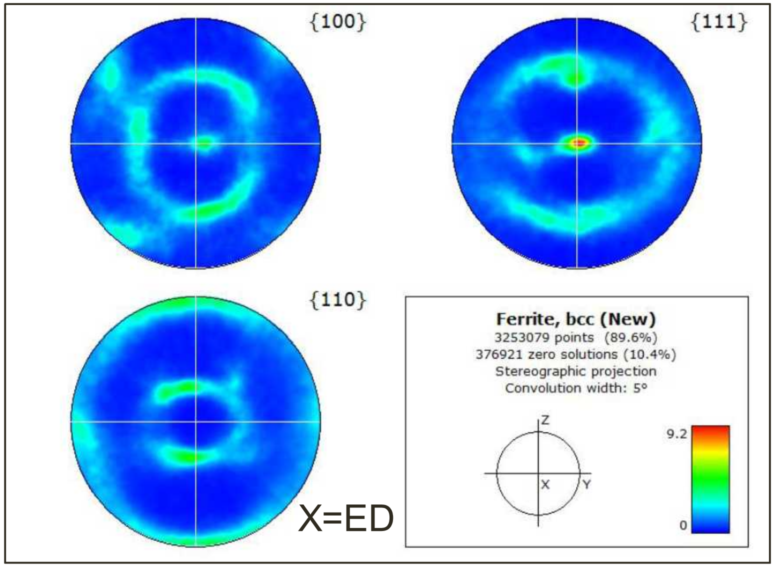


Fig. 2. X-ray diffraction results displaying the texture of NbZr in the as-received (CG), route-8B$_C$-processed and route-16E-processed states. Equal-area projection inverse pole figures show the data for extrusion (3), longitudinal (2) and flow (1) directions. The intensity scales are logarithmic.

T. Niendorf, D. Canadinc, H.J. Maier, I. Karaman, G.G. Yapici, *Acta Mat*, Vol 55, (2007),6596-6605.



Hiperco HCO1: **Route E**
4 passes 850C, WQ

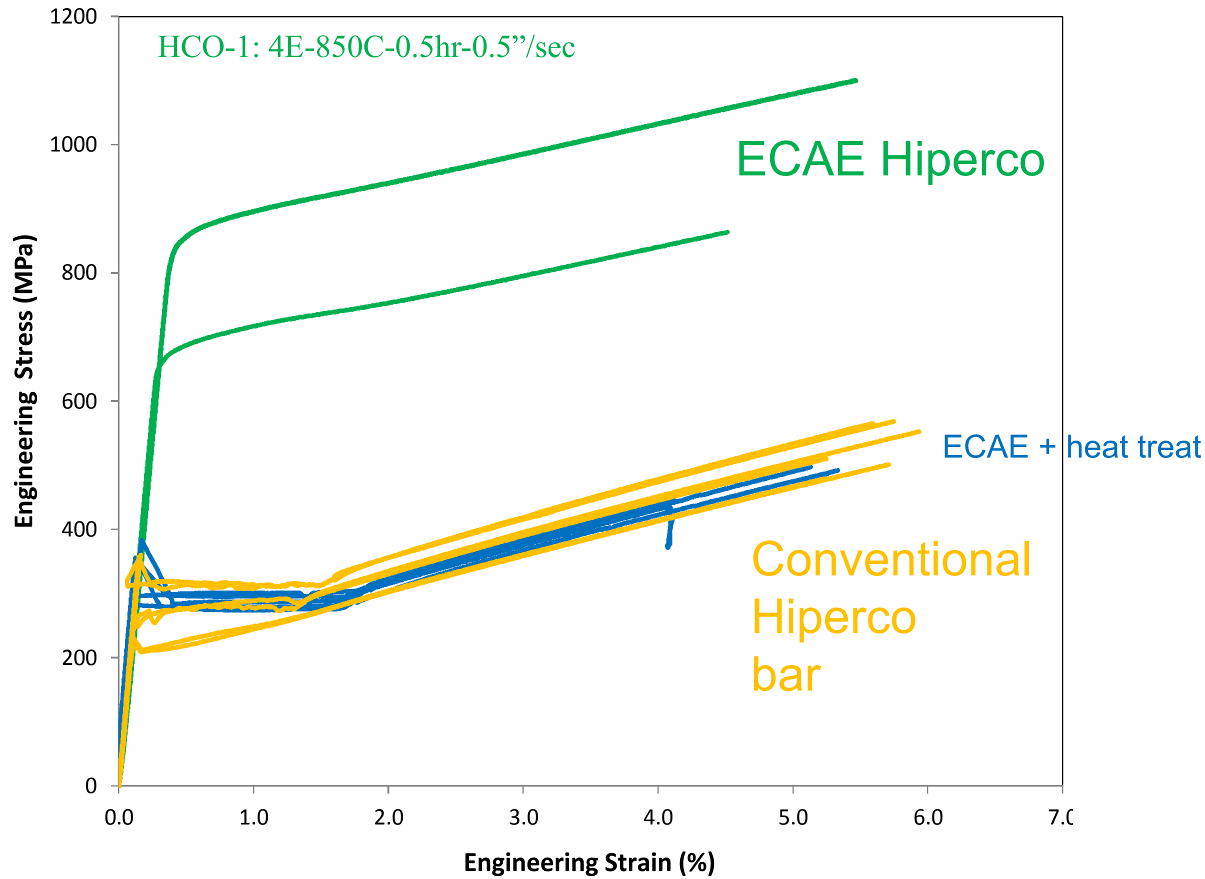


Hiperco HCO5: **Route E**
4 passes 850C, 2 passes 750C WQ

High Strength Achieved

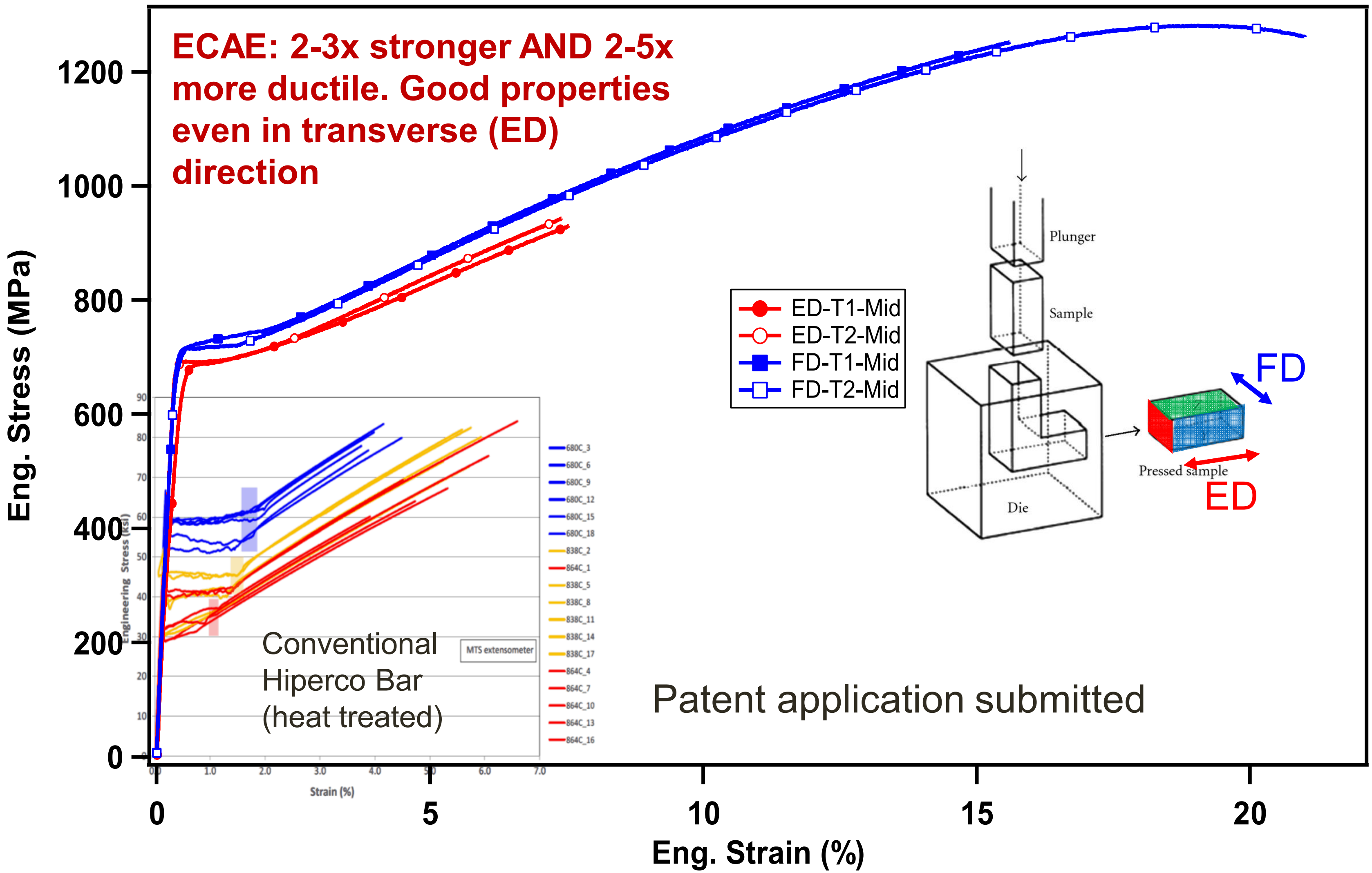
HCO-1, 4 passes, Route E, 850C

- 2-3 fold increase in strength in the as-ECAE condition
- Ductility is comparable to conventional Hiperco bar
- Heat treatment (to gain magnetic performance) results in low strength comparable to conventional bar



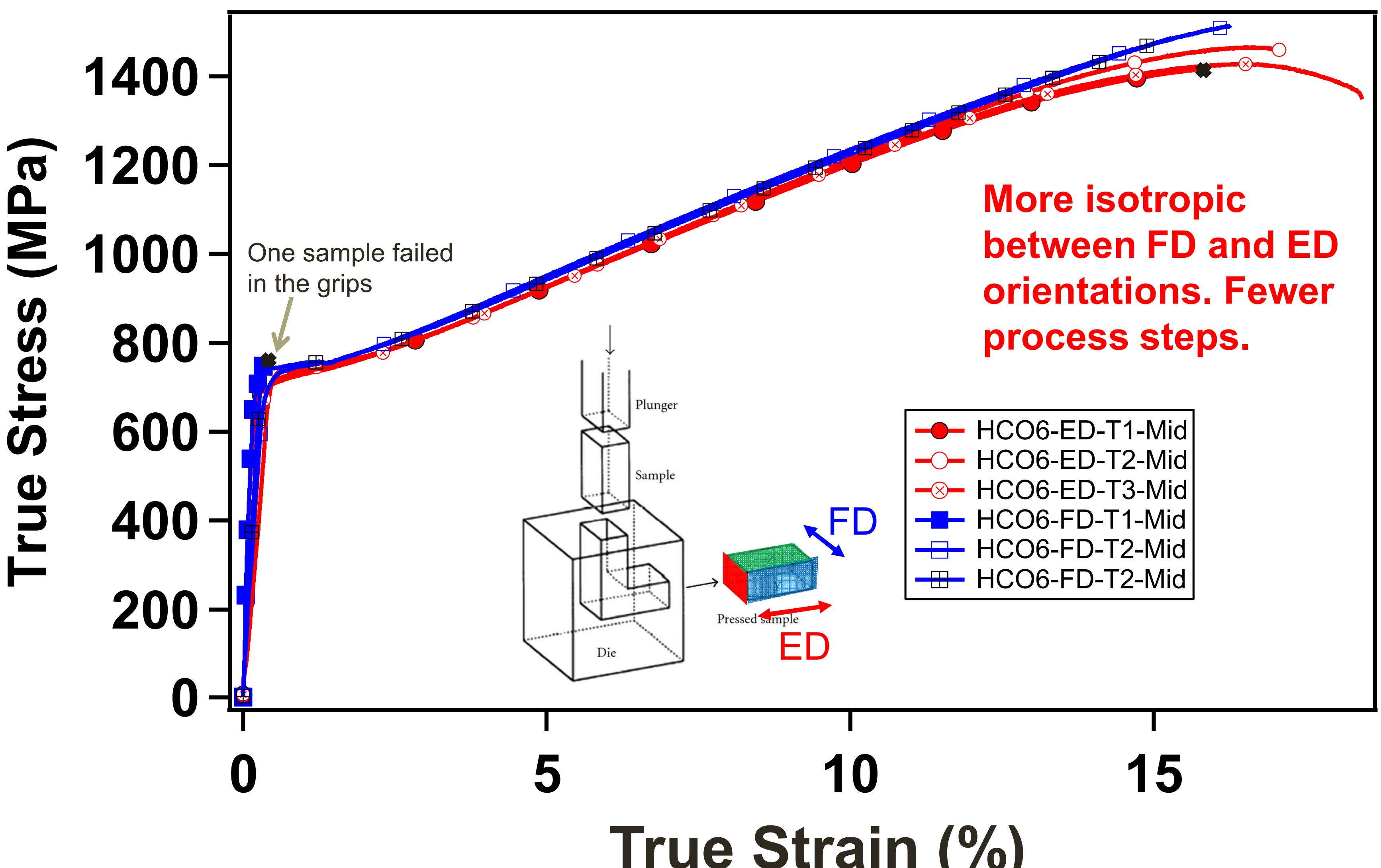
High Strength AND High Ductility

HCO-5: 4E 850°C +2E-750°C



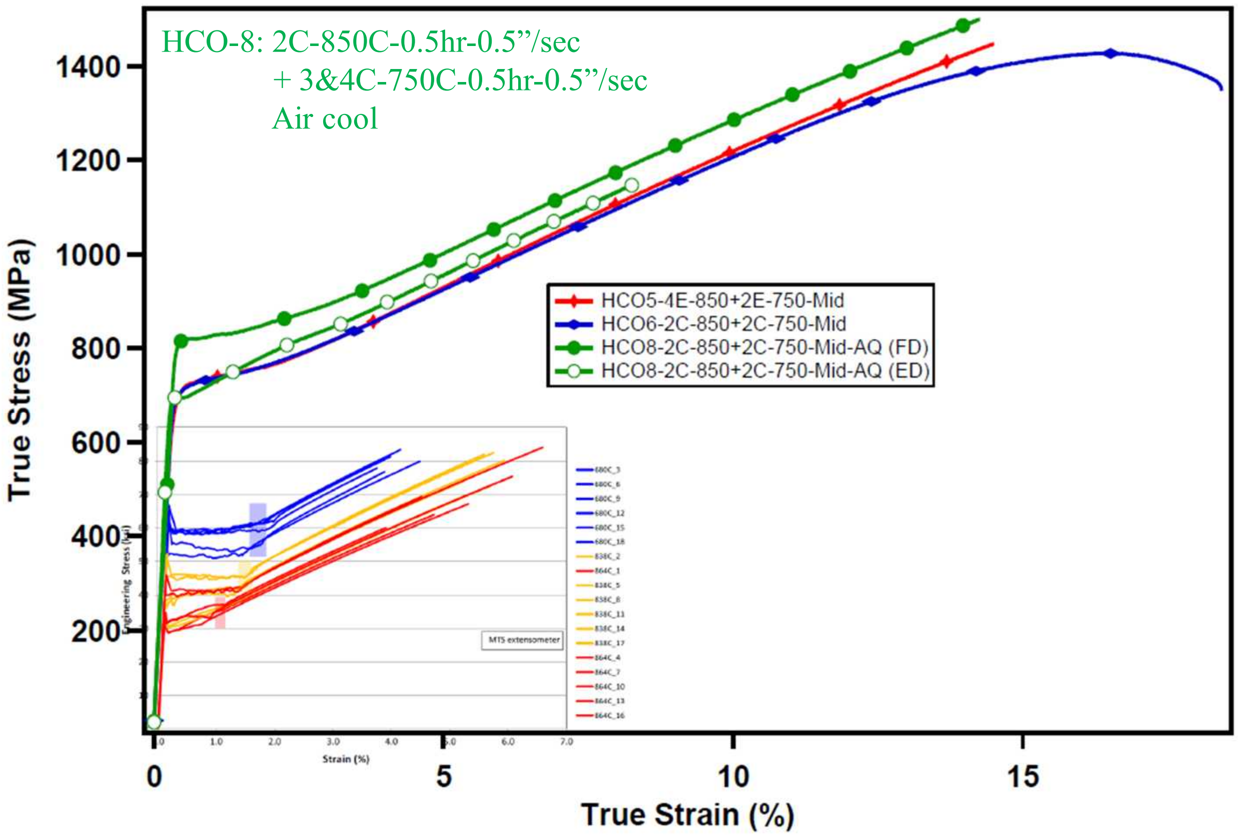
More Isotropic with Fewer Extrusion Passes

HCO-6: 2C 850°C +2C 750°C



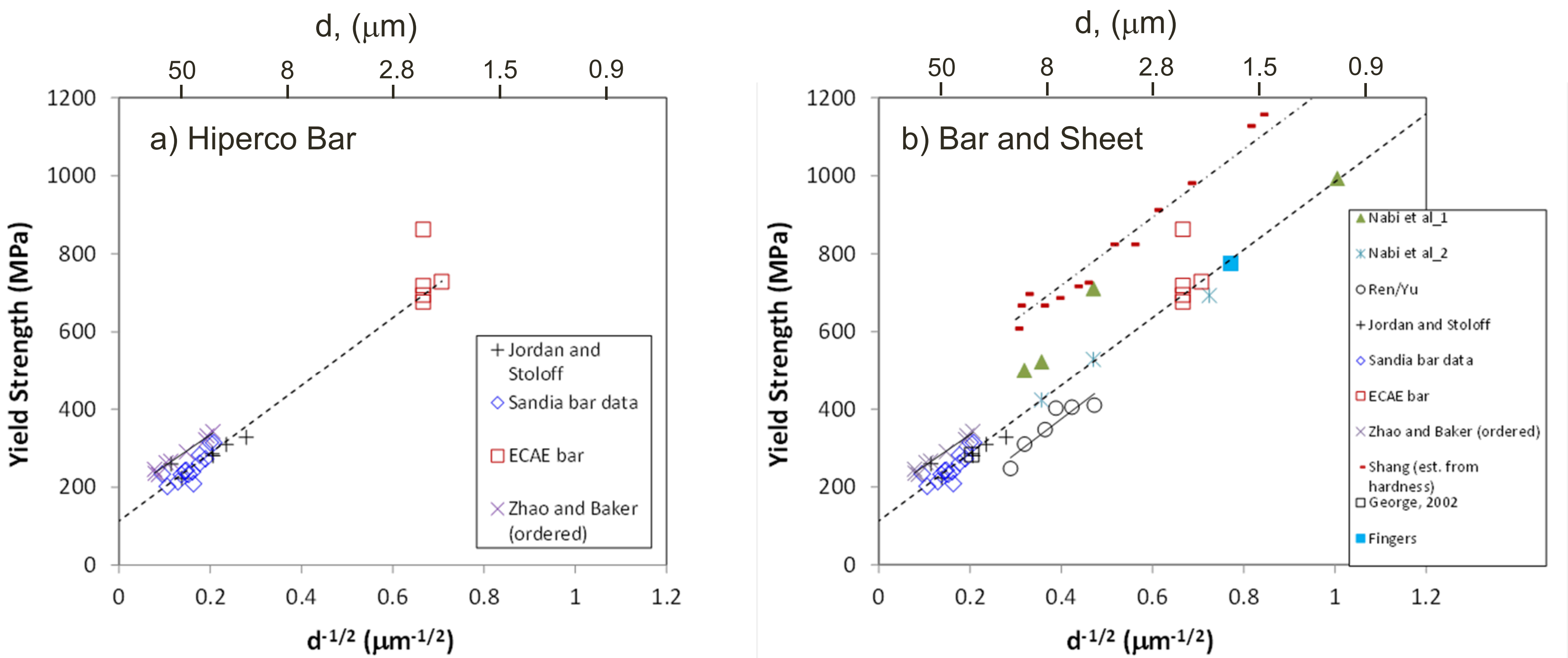
Effects of Air-Cooling after ECAE

HCO-8: 2C 850C + 2C 750C, Air Cool



- HCO-8: Same as HCO-6, except air cool after ECAE
- Attempt to produce ordered B2 crystal structure *without separate heat treatment*
- Still needs to be characterized for magnetic performance, B-H rings have been machined from HCO-11

Strength follows Hall-Petch Relationship



- Highest strength Hiperco ever produced in *BAR form (As-ECAE condition)*
- Strength comparable to fine-grain, cold-worked sheet

Follows Hall-Petch relationship: $\sigma_y = \sigma_o + kd^{-1/2}$

σ_y: yield strength, σ_o: intrinsic (friction) stress,

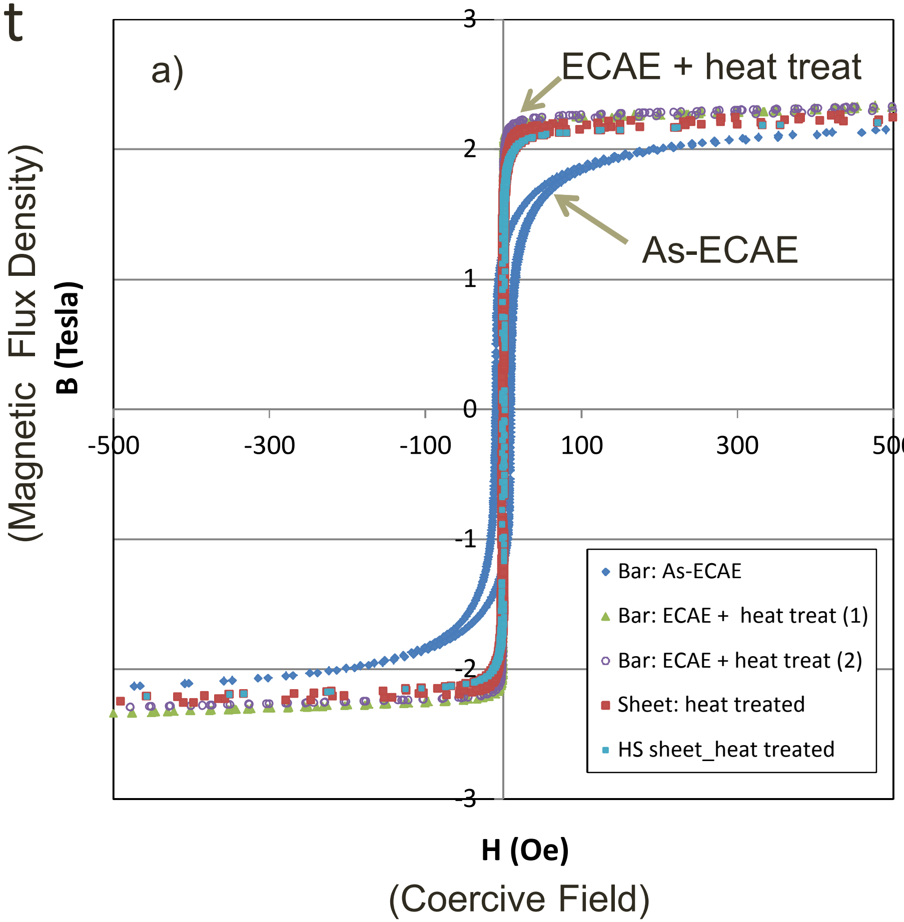
k: constant (Hall-Petch coefficient), d: grain size

Magnetic Performance of ECAE Material

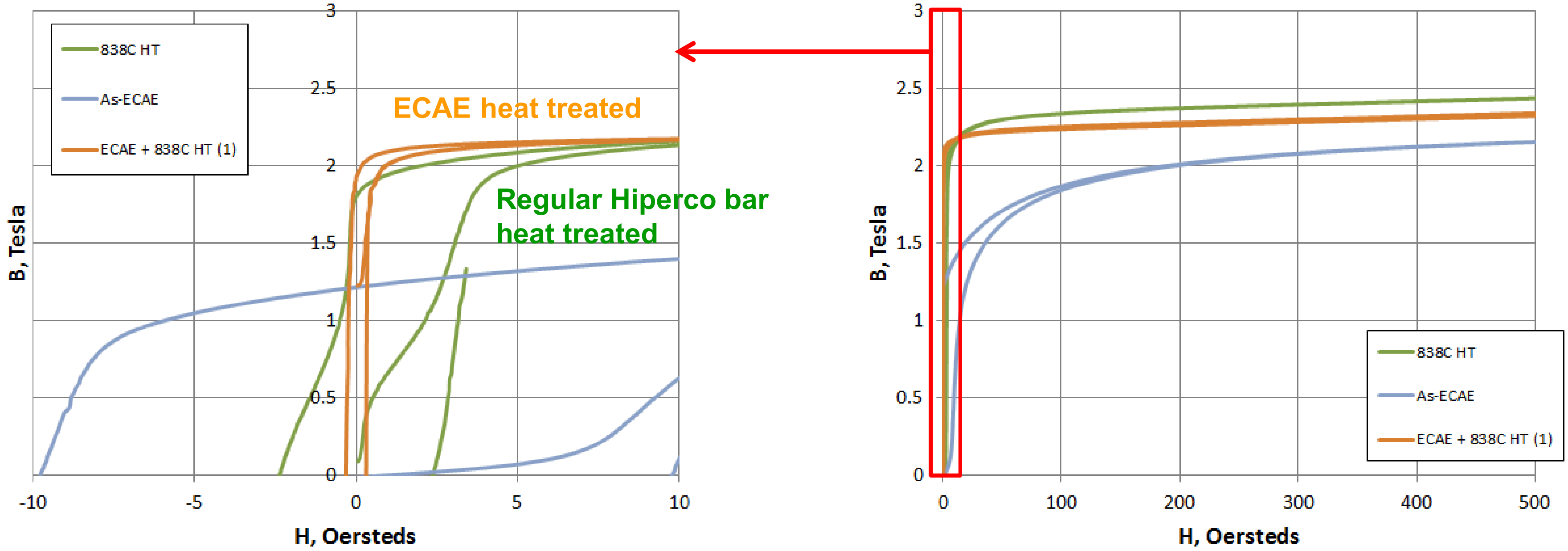
- Magnetic B-H test rings machined from ECAE material and tested according to ASTM A773
- Compared to Hiperco sheet

- Heat treatment after ECAE results in very good magnetic response. Saturation (B) comparable to, or higher than, Hiperco sheet

- Unfortunately, mechanical properties are sacrificed in the fully heat treated condition



Excellent Magnetic Performance of ECAE Material in Heat Treated Condition

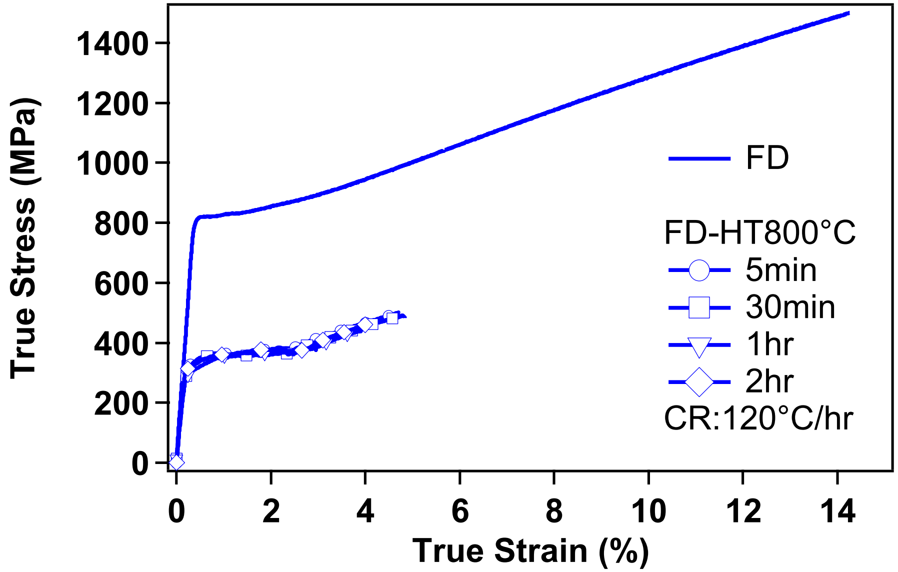


- Heat treatment after ECAE results in very good magnetic response.
- Unfortunately, mechanical properties are sacrificed in the fully heat treated condition

Material	Bmax (Tesla)	Br (Tesla)	Hc (Oersteds)	µmax
As-ECAE Hiperco	2.15	1.21	9.78	723
Heat treated ECAE Hiperco	2.34	1.82	0.30	~37000
Conventional bar_838C HT	2.47	1.92	2.35	28283

Ongoing Efforts

- Investigate alternative heat treatments after ECAE. Will *short times at lower temperatures* give good magnetic performance AND good mechanical response?
- Magnetic measurements on air-cooled material
- Characterization of grain size and crystallographic texture
- Repeat tensile testing on recent extrusions -- show repeatability



Even only 5 minutes at 800C is enough to eliminate ECAE structure and reduce mechanical properties

Currently investigating lower temperature heat treatments

Summary:

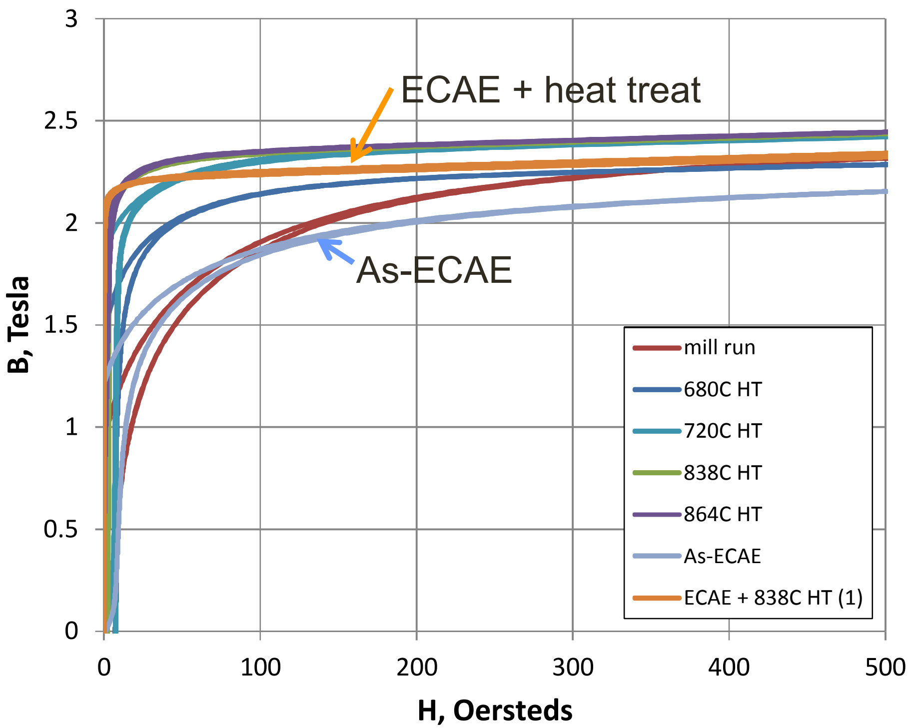
- ECAE can be successful in the 850 to 750°C range
- Significant microstructural refinement is achieved
- Full heat treatment (for magnetic properties) reduces strength and ductility to conventional bar levels
- HCO-5 and HCO-6 (850C followed by 750C): High strength AND high ductility. Why?? Suggests that 750C limits recrystallization in favor of deformation (cold work)…points toward a dislocation substructure being important for ductility.
- Magnetic behavior is “moderate” in the as-ECAE condition, very good in the heat treated condition.

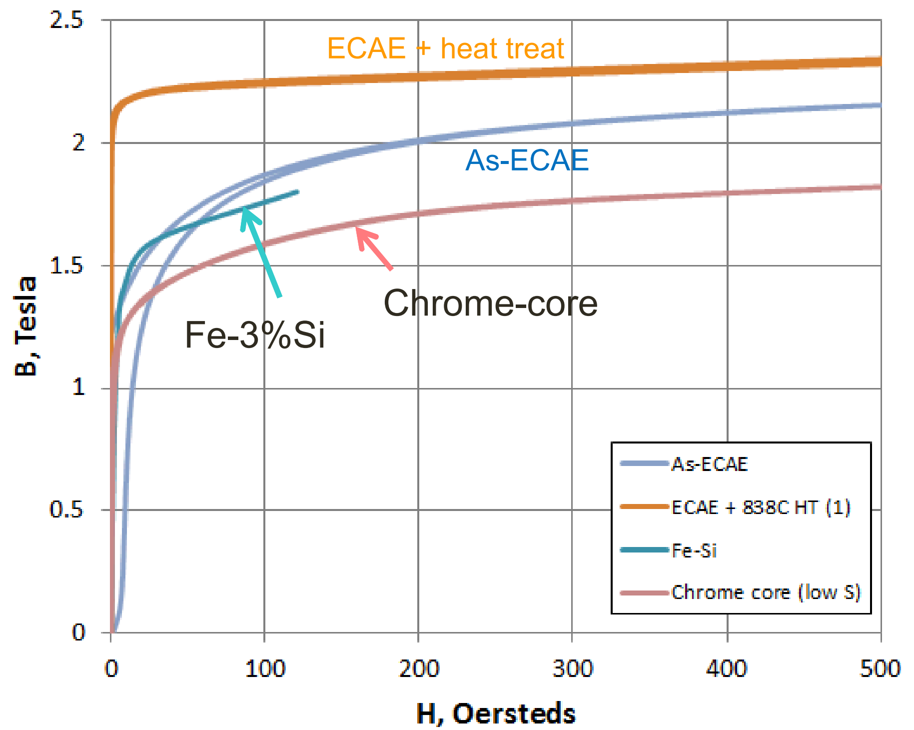
Future Work:

- Explore other bar diameters for ECAE...commercial vendor? Longer billet lengths, larger diameters can be produced commercially.
- Address the issue of surface cracking. Clad with Cu sheet or spray on Cu prior to ECAE?
- Investigate notch sensitivity and surface finish effects on Hiperco
- Develop a capability to measure “order parameter” (degree of order) in Hiperco

- Extra slides

Magnetic Performance, other materials





<u>Compared to conventional Hiperco bar</u>

- As extruded condition comparable to "mill run" Hiperco
- In heat treated condition, magnetic saturation is slightly lower, permeability slightly higher than conventional bar

<u>Compared to other soft magnetic alloys</u>

- Competes well with other alloys, even in the as-ECAE condition