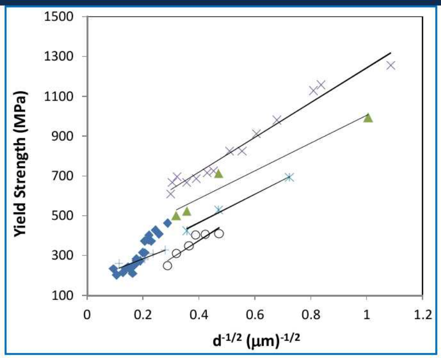
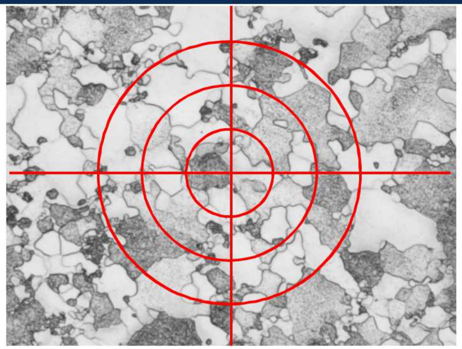
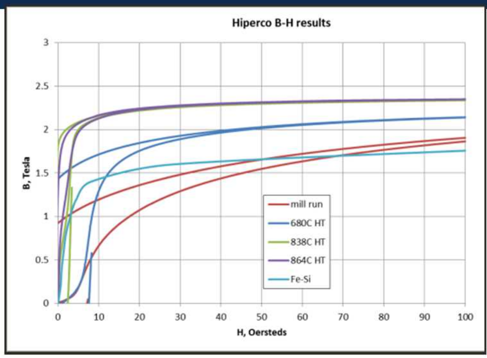
SAND2019-2484PE







Advanced Processing of Fe-Co-2V “Hiperco” Soft Magnetic Alloy

Don Susan and Jeff Rodelas

Sandia National Laboratories

Seminar, Colorado School of Mines

March 7, 2019

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-NA0003525.

Collaborators

Sandia: Mark Reece, Dan Tung, Mike Maguire, Jay Carroll, Andrew Kustas, Rick Kellogg, Sara Szarka, Joe Michael, Sara Dickens, Joe Michael, Jay Carroll, Alex Barr, Matt Vieira

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

Shear-Form Inc.: Bryan, TX

Topics

Introduction and History

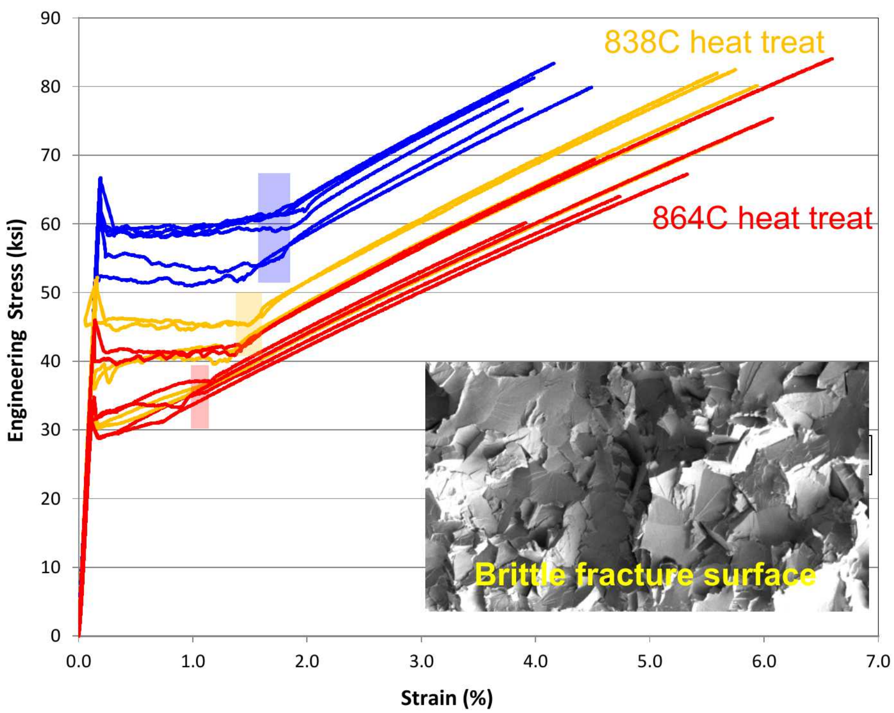
Equal Channel Angular Extrusion (ECAE)/Pressing (ECAP)

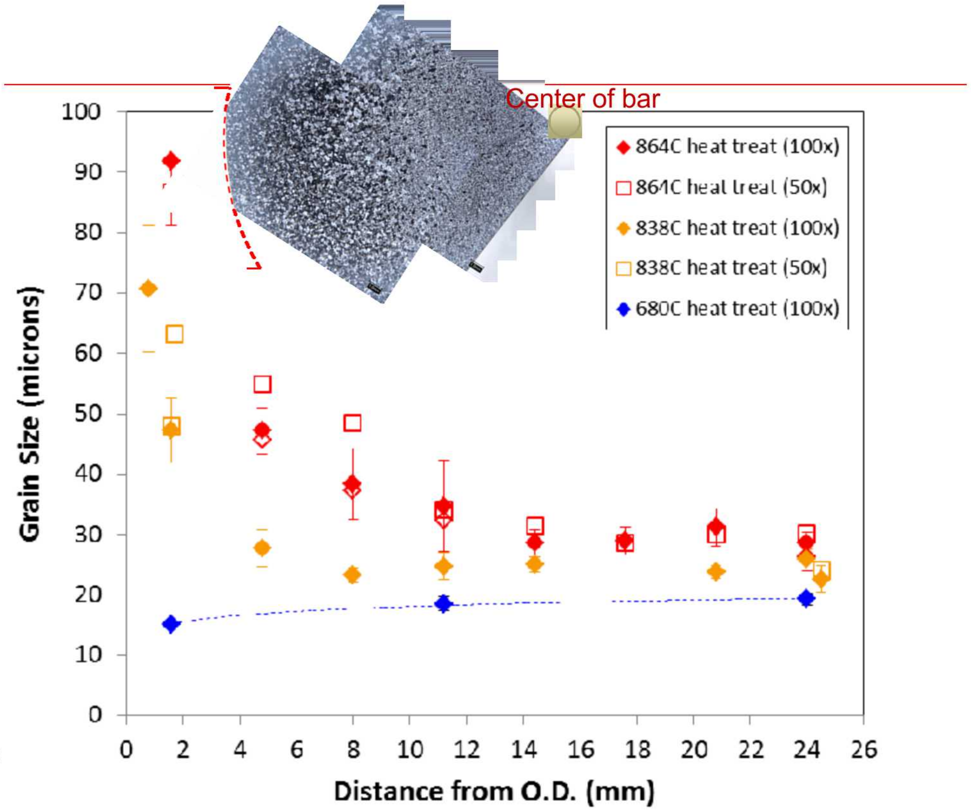
Solid-State Welding (Inertia Welding) of Hiperco to other alloys

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 alloy system
- Used in solenoids, electric motors, and other components





Why does Hiperco have poor mechanical properties?

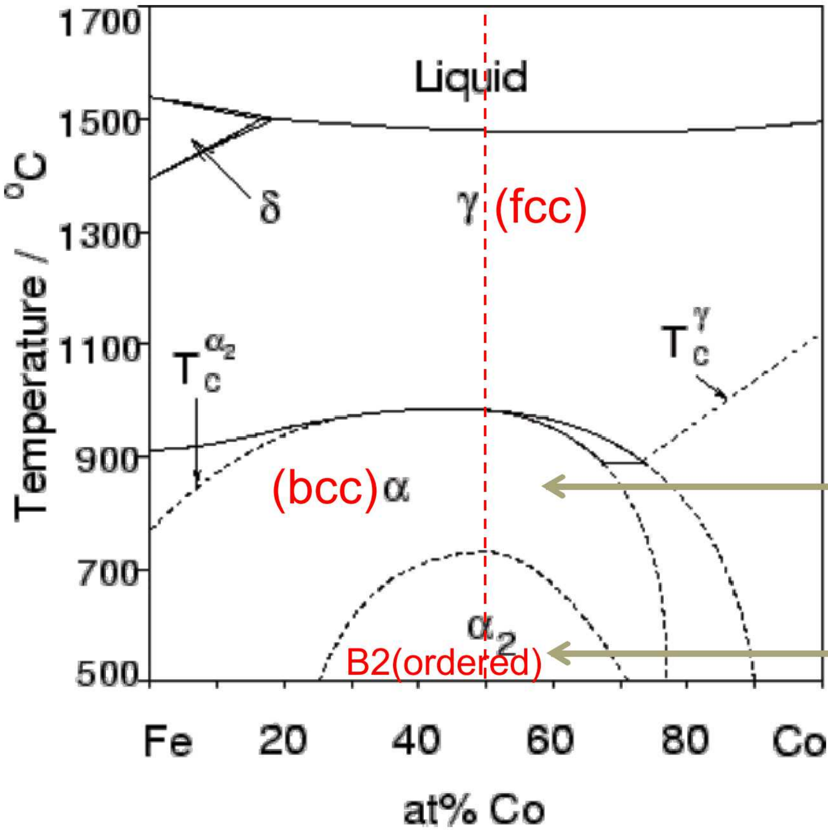


Figure: The Fe-Co binary diagram as given in [5]. T_c denotes the Curie temperature.

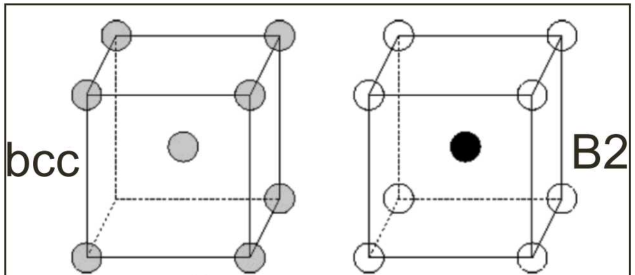


Figure: b.c.c. and ordered B2 structure.

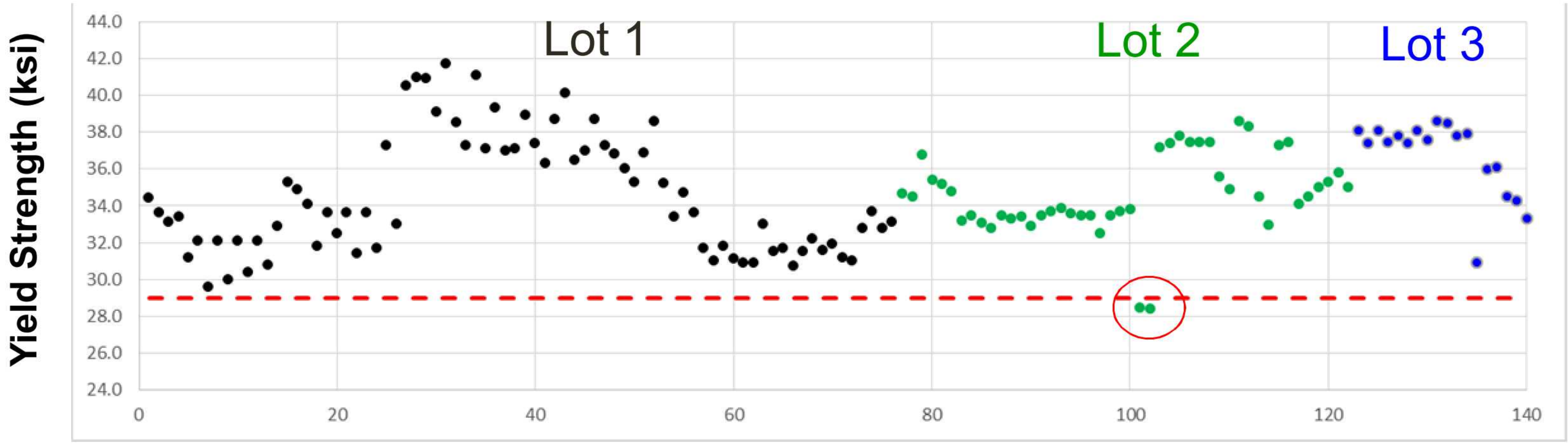
Crystal structures of Hiperco alloy

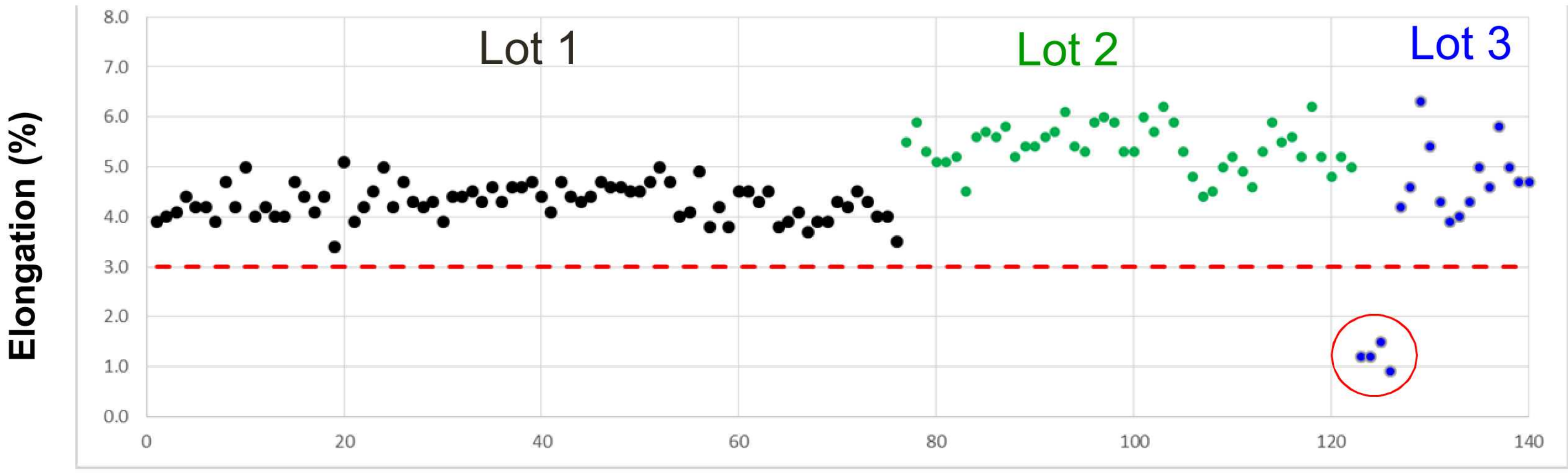
bcc: Good mechanical properties, OK magnetic properties

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

History: Marginal Mechanical Properties



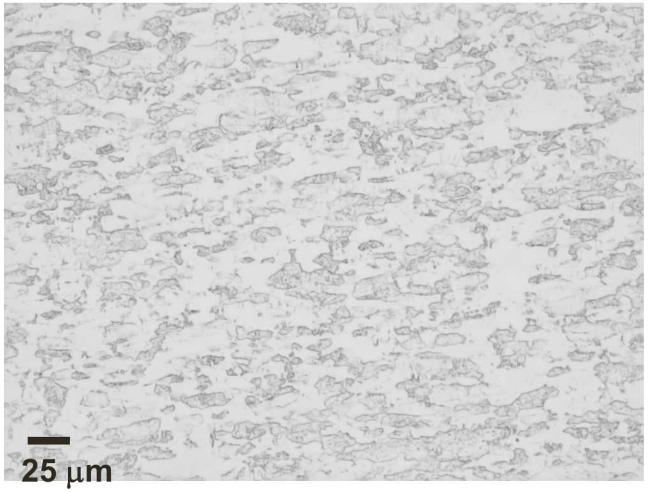


Comparison to Sheet

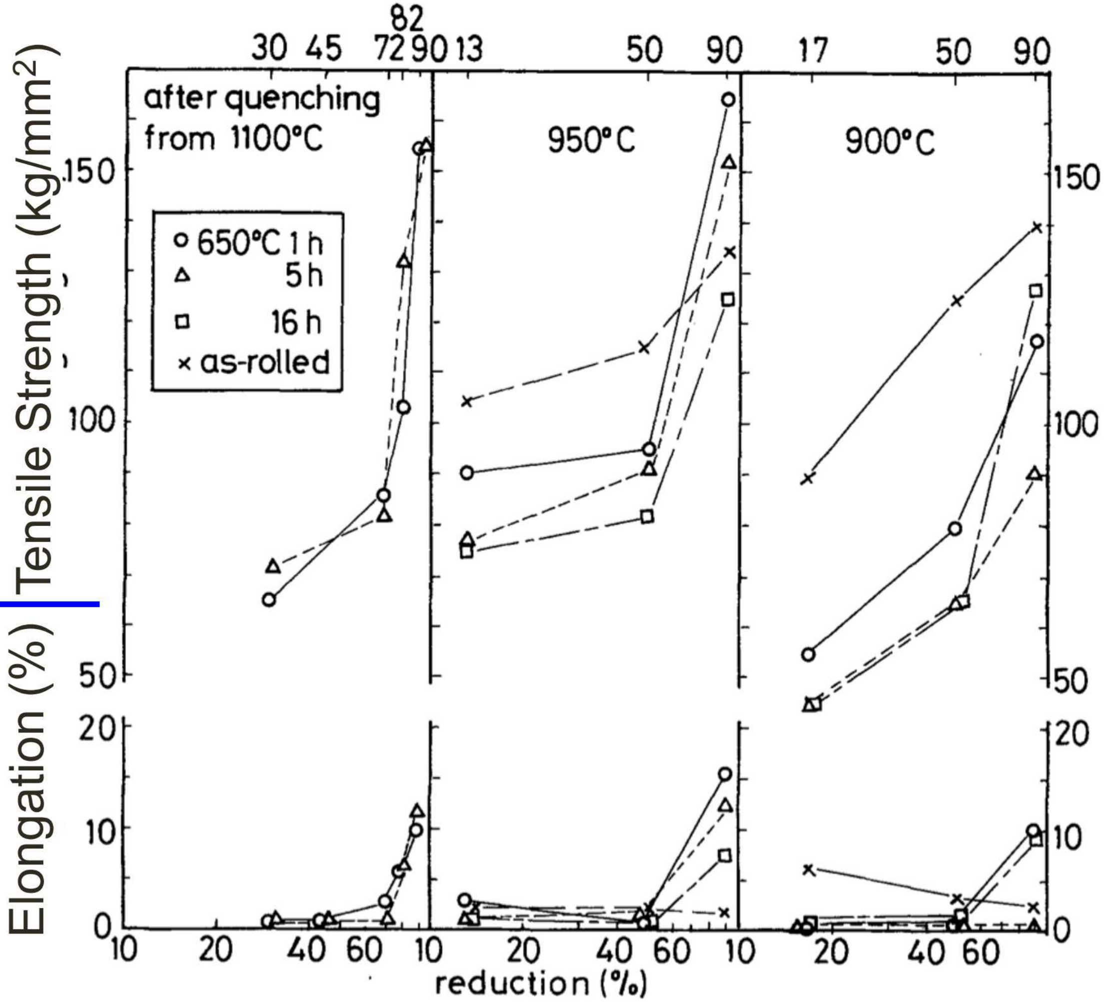
Hiperco rolled bar is weak, brittle, and inhomogeneous. In contrast, *sheet rolled* Hiperco is stronger and can be more ductile, with adequate magnetic performance.

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

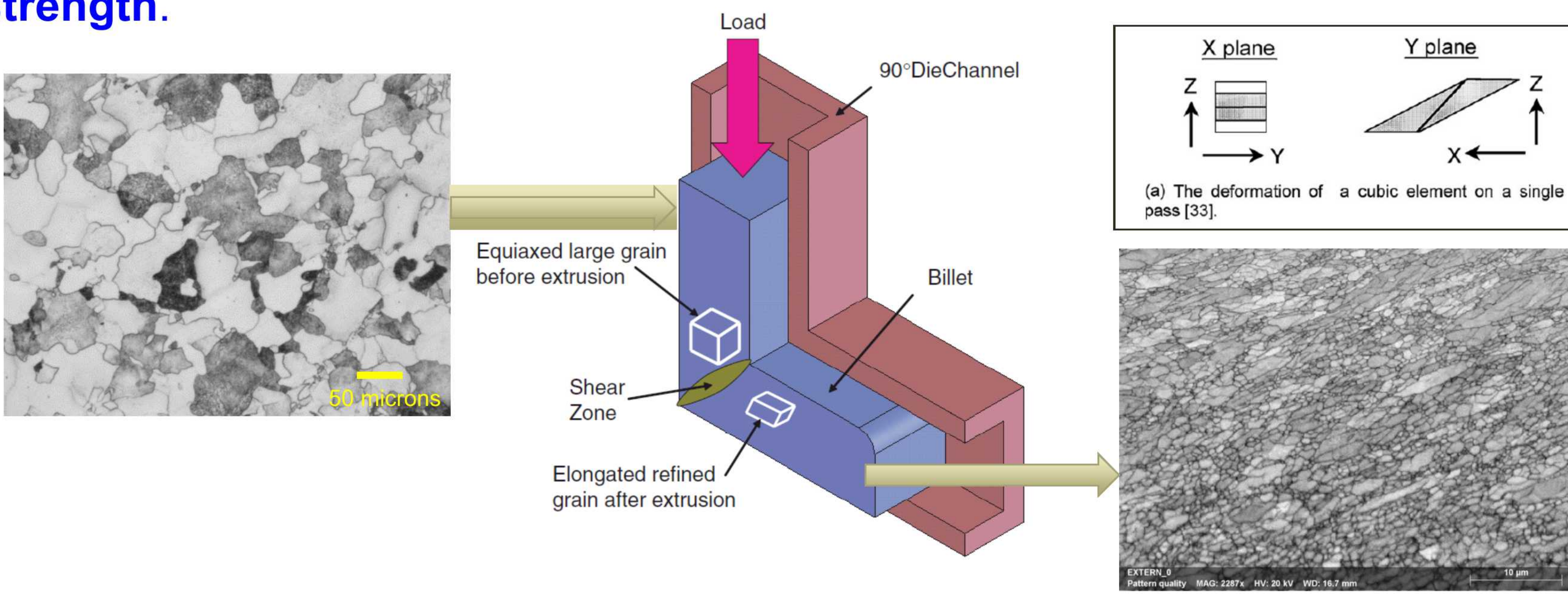


Equal Channel Angular Extrusion (ECAE), a.k.a. Equal Channel Angular Pressing (ECAP)

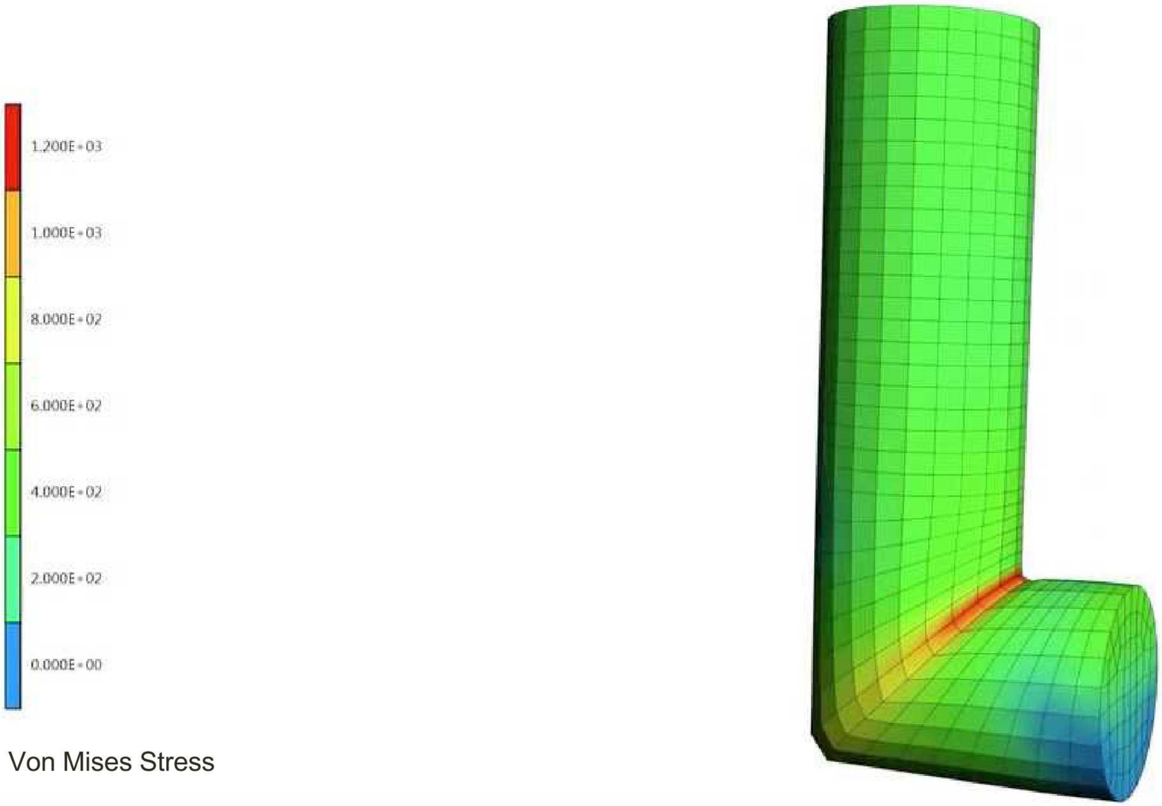
The premise: High performance Hiperco magnetic components would benefit greatly from increased mechanical strength and/or ductility.

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**.

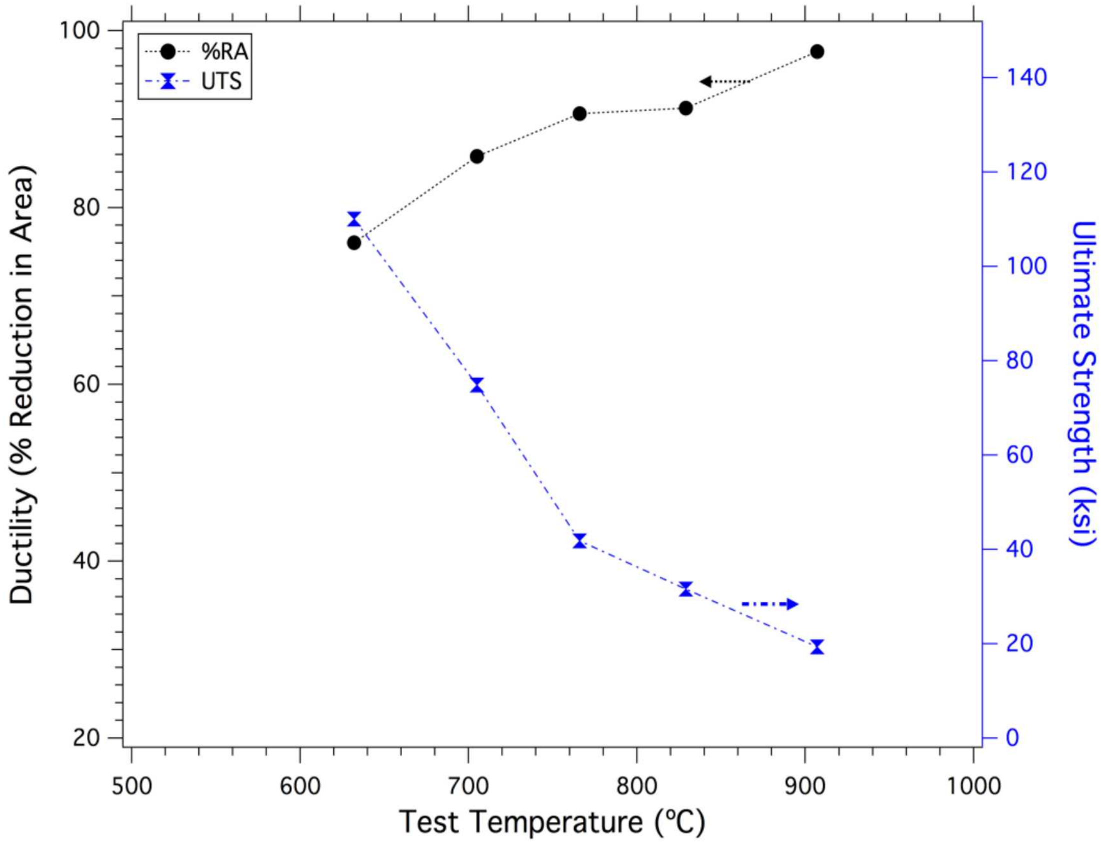


Equal Channel Angular Extrusion/Pressing



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.

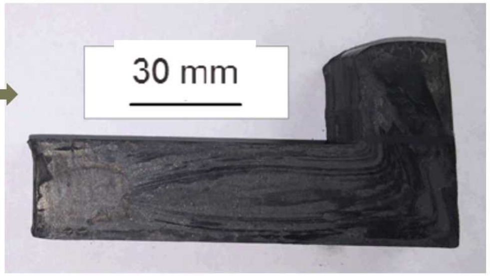
High-Temperature Ductility of Hiperco



- 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

18 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, tensile tests complete
HCO-10	1&2C-850C / 3&4C-750C (WQ)	Same as HCO6
HCO-11	1&2C-850C / 3&4C-750C (AC)	Same as HCO8, magnetic tests complete
HCO-12	1 pass, 850C (WQ)	
HCO-13	2C - 850C (WQ)	
HCO-14	2C 850C / 1C 750C (WQ)	
HCO-15	2C 850C / 2C 750C (AC)	Same as HCO-8, HCO-11
HCO-16	2E 850C (WQ)	
HCO-17	2E 850C / 1E 750C (WQ)	
HCO-18	2E 850C / 2E 750C (AC)	Same as HCO-5, except air cool

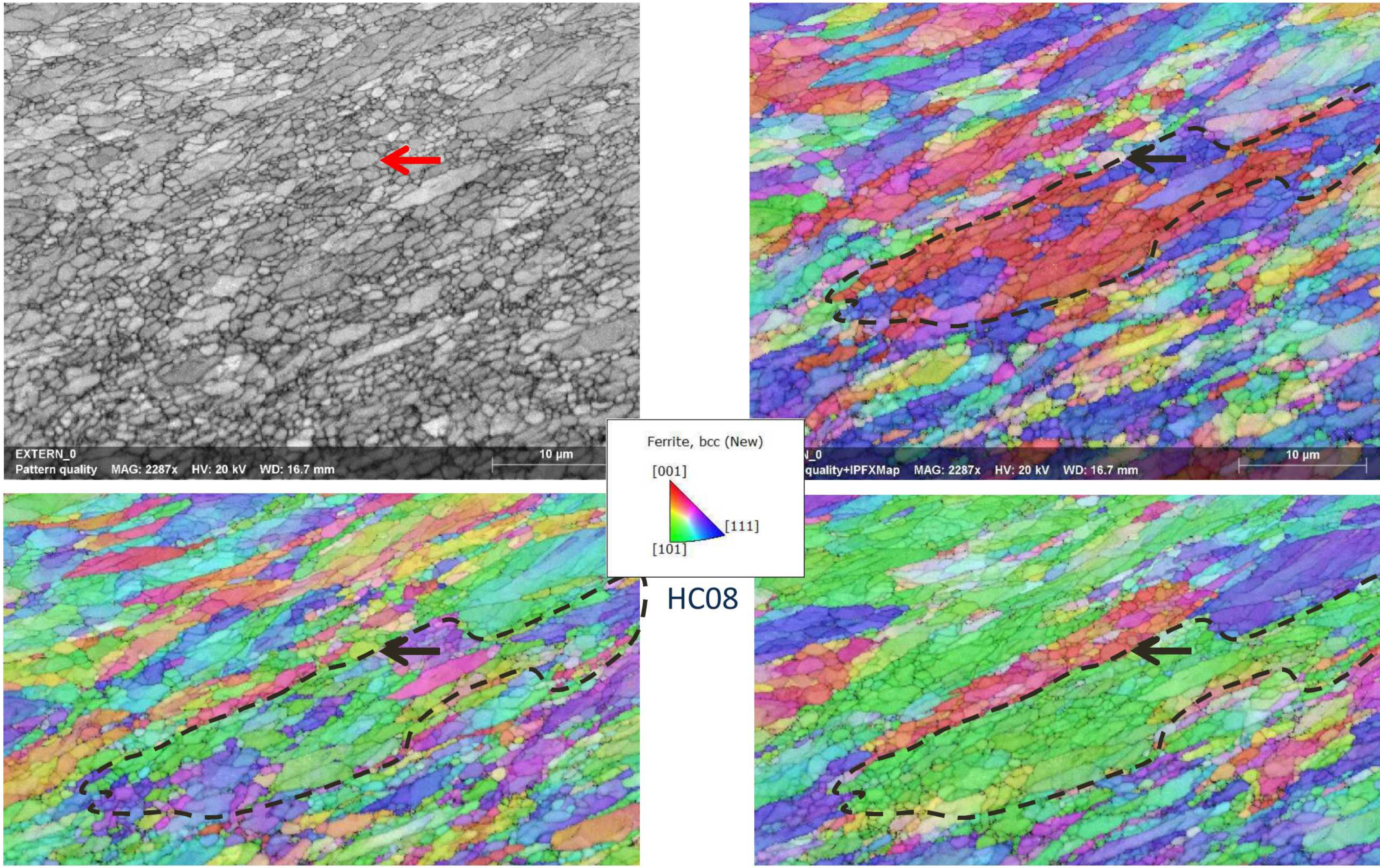


Results show ECAE at 850 → 750°C range can successfully produce material for testing

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

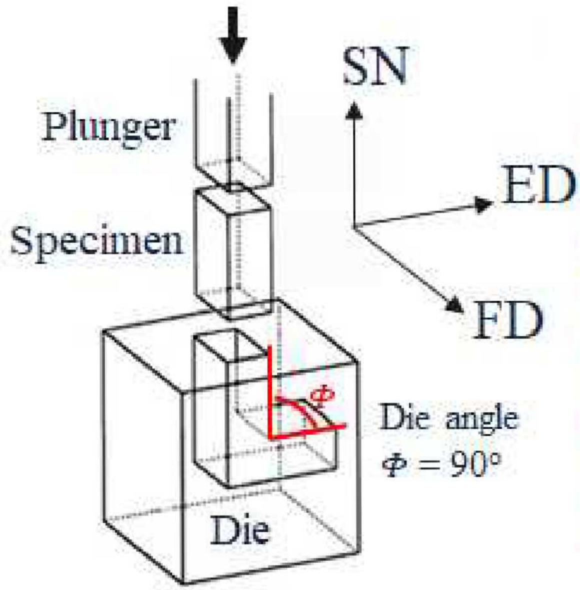
Route name	Min. # of passes	Billet rotations about the extrusion axis				Material Yield*	Effect on microstructure
		1 →	2 →	3 →	4 → N		
A	1	0°	0°	0°	etc.	0.58	elongation (lamellar)
B (B_A)	2	+90°	-90°	+90°	etc.	0.67	elongation (filamentary)
C	2	180°	180°	180°	etc.	0.83	back/forth shearing
C' (B_C)	4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

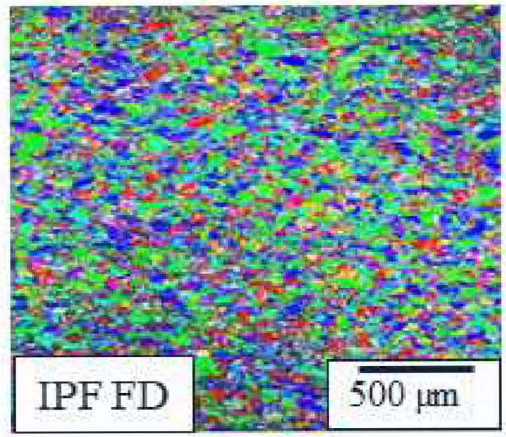
EBSD/Texture Analysis



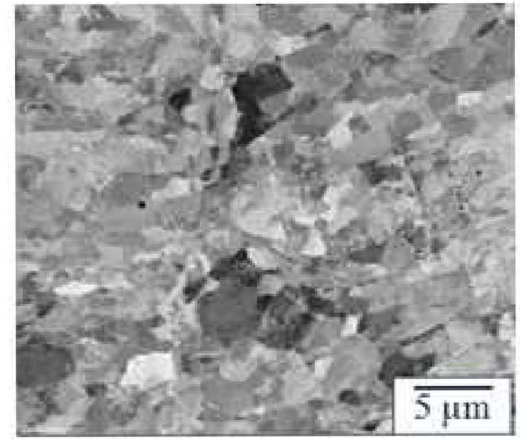
- Significant grain refinement achieved (2-3 microns compared to 25-50 for regular bar)
- Uniform microstructure from center to edge of billet
- Note, this Hiperco lot has 600 ppm Nb (meant to promote grain refinement)

Texture of ECAE Material



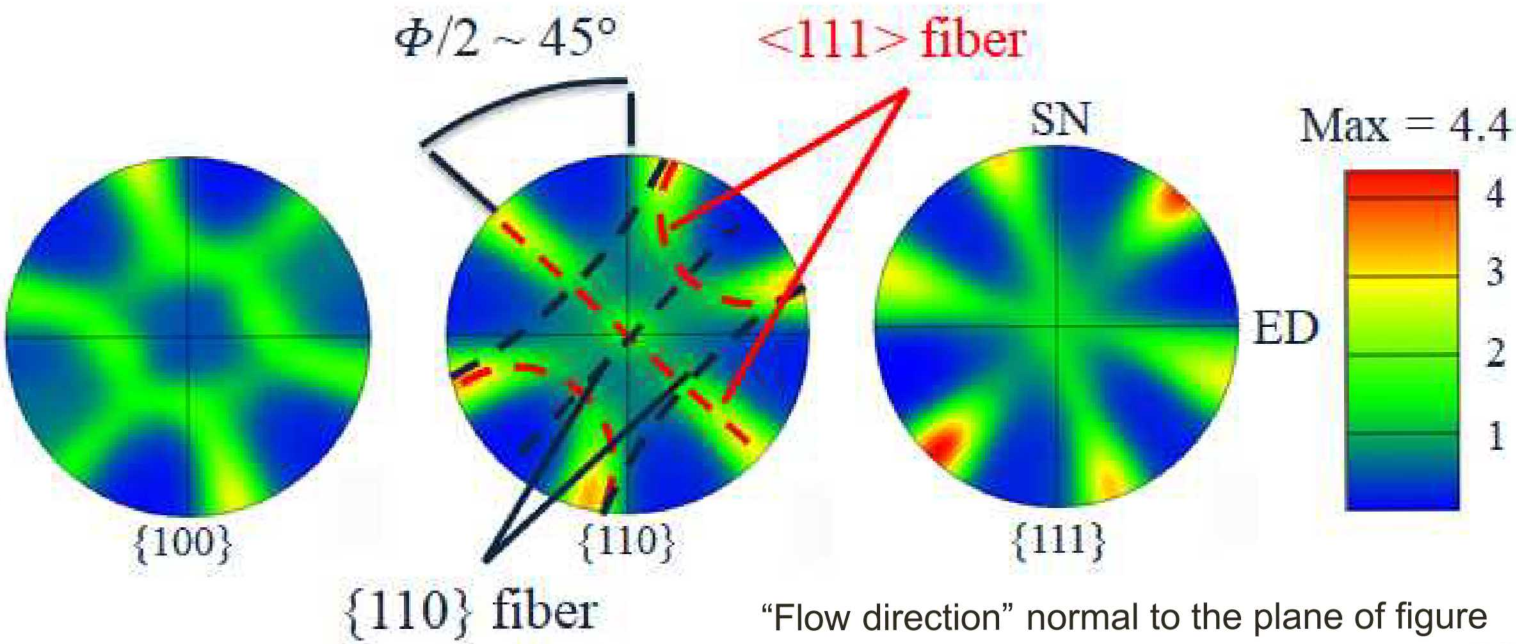


Approx. 2 micron grain size



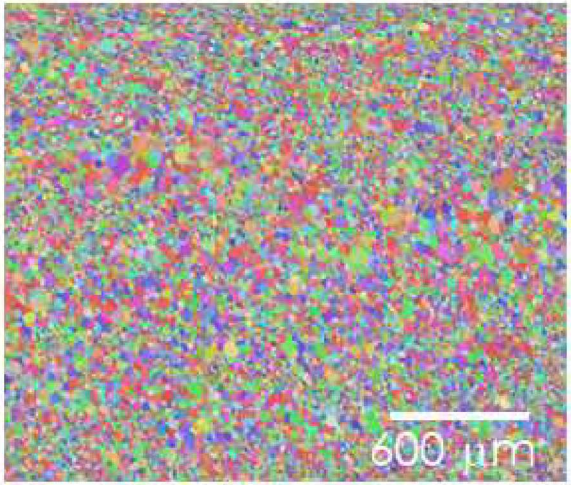
Single pass ECAE: "simple shear" texture, dual partial {110} and <111> fibers

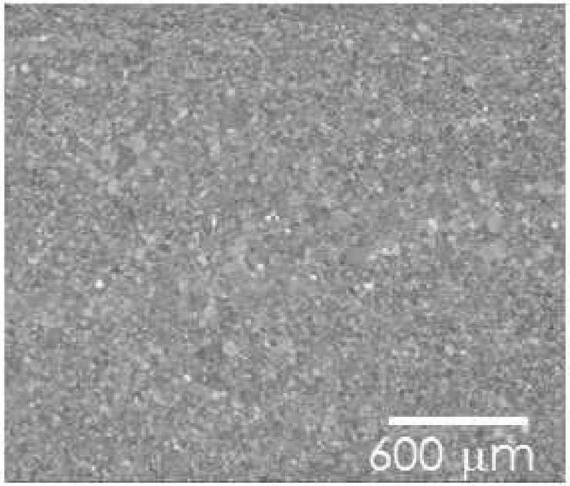
Inclined relative to the bar axis due to ECAE shear

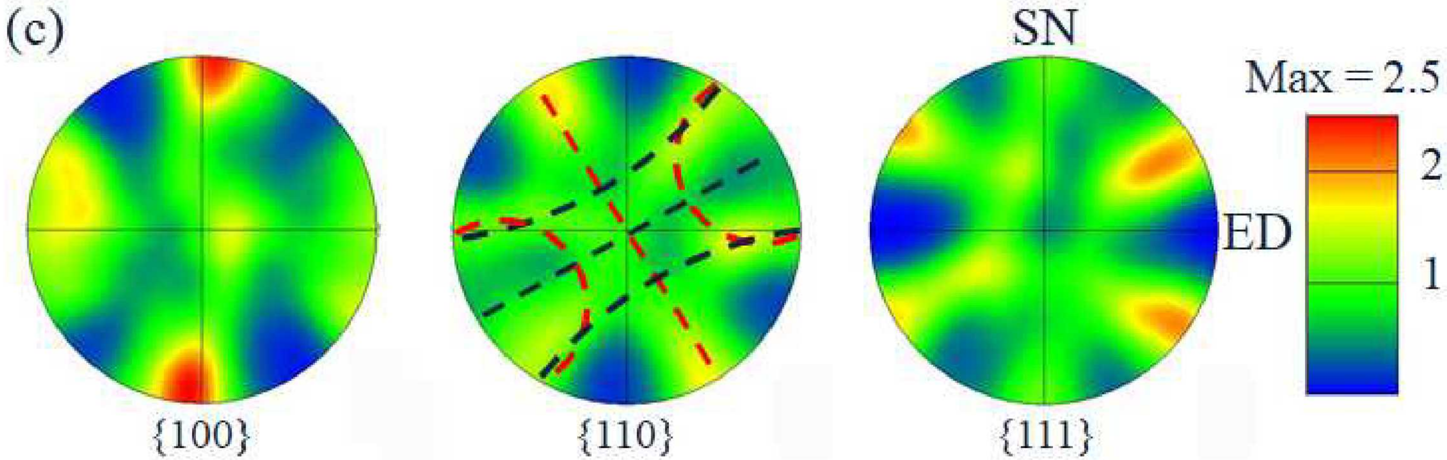


"Flow direction" normal to the plane of figure

Texture of Post-ECAE Annealed Bar



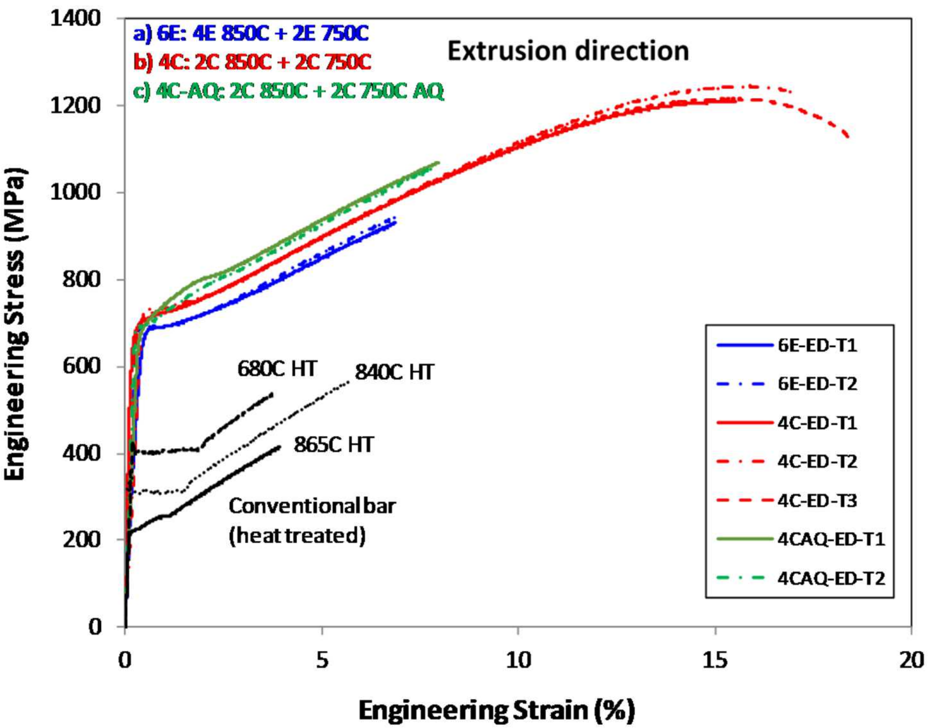


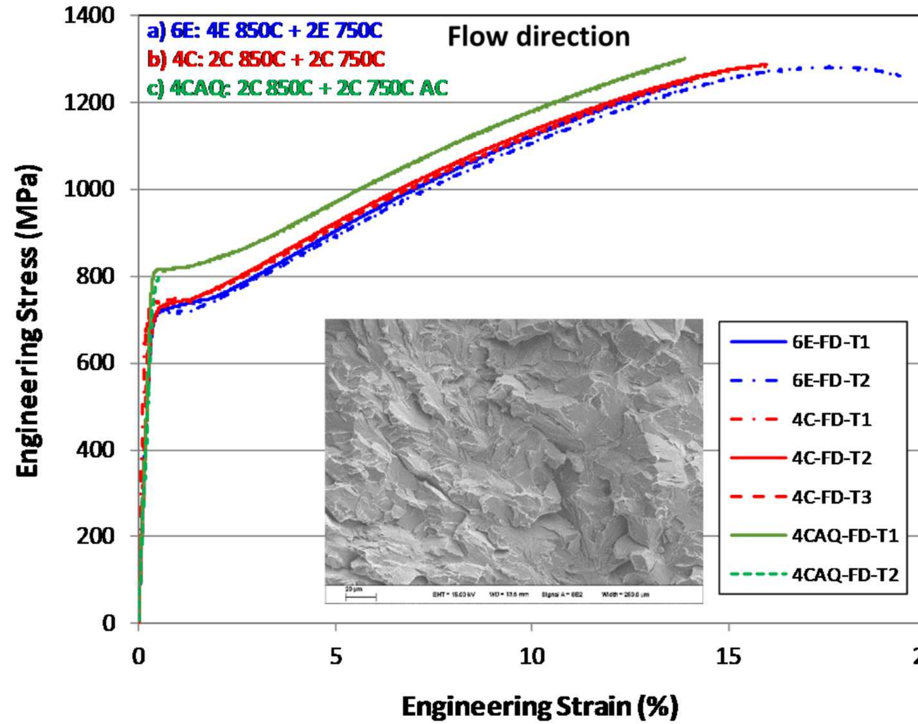


Texture remains the same, although somewhat weakened by post-ECAE heat treatment (necessary for magnetic property development). Significant grain growth compared to as-ECAE condition.

High Strength AND High Ductility are Achieved in AS-ECAE Condition

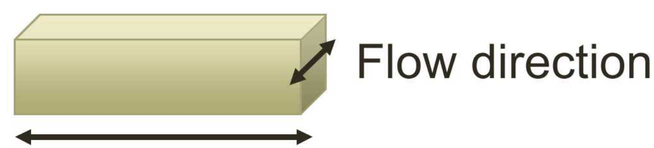
U.S. Patent Pending



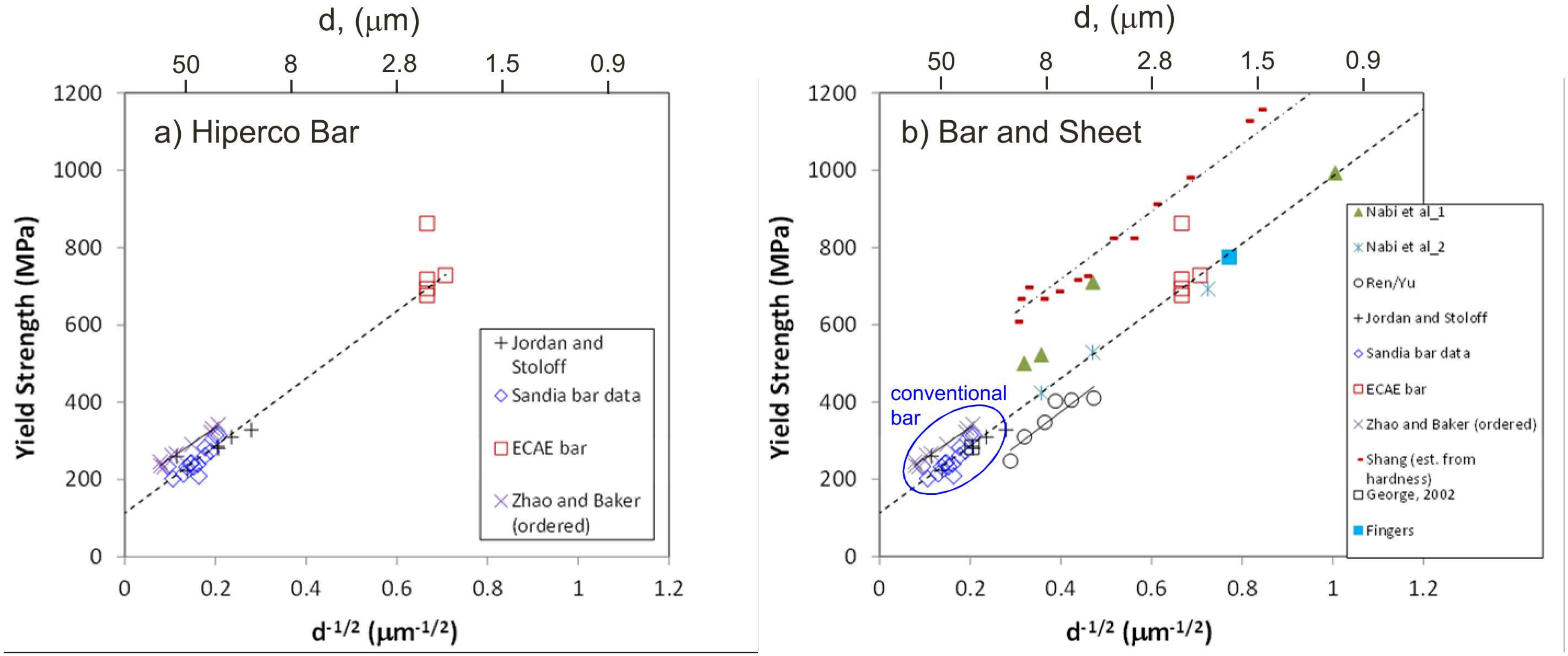


ECAE: 2-3x stronger AND 2-5x more ductile (primarily due to disordered structure). Good properties even in transverse (FD) direction.

Note: Route C appears more isotropic between FD and ED orientations. Fewer process steps.



Strength follows Hall-Petch Relationship



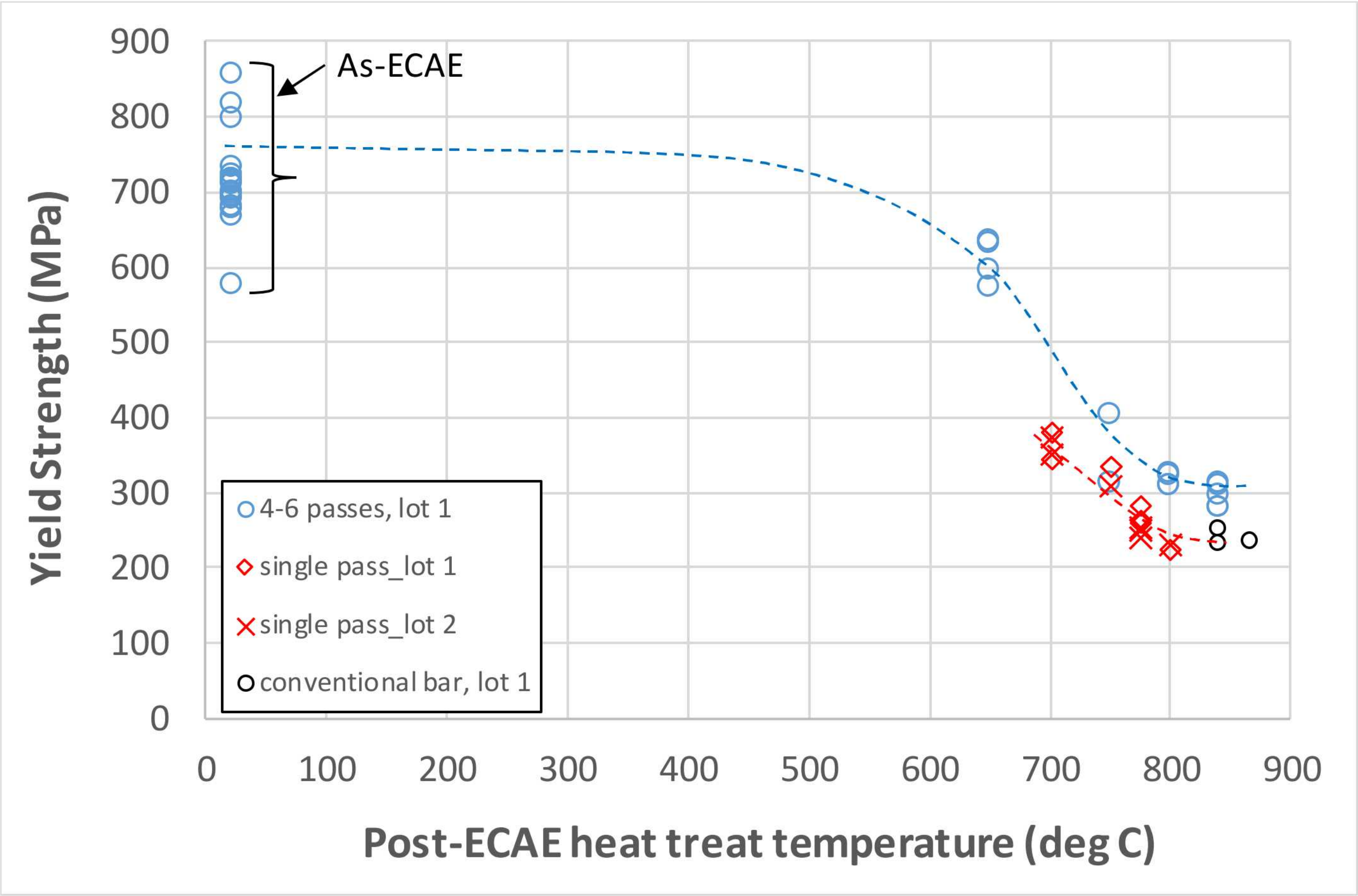
- Highest strength Hiperco ever produced in *BAR form (As-ECAE condition)*
- Strength comparable to fine-grain, cold-worked sheet

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

Recent Post-ECAE Optimization Trials



840°C is standard heat treat temperature.

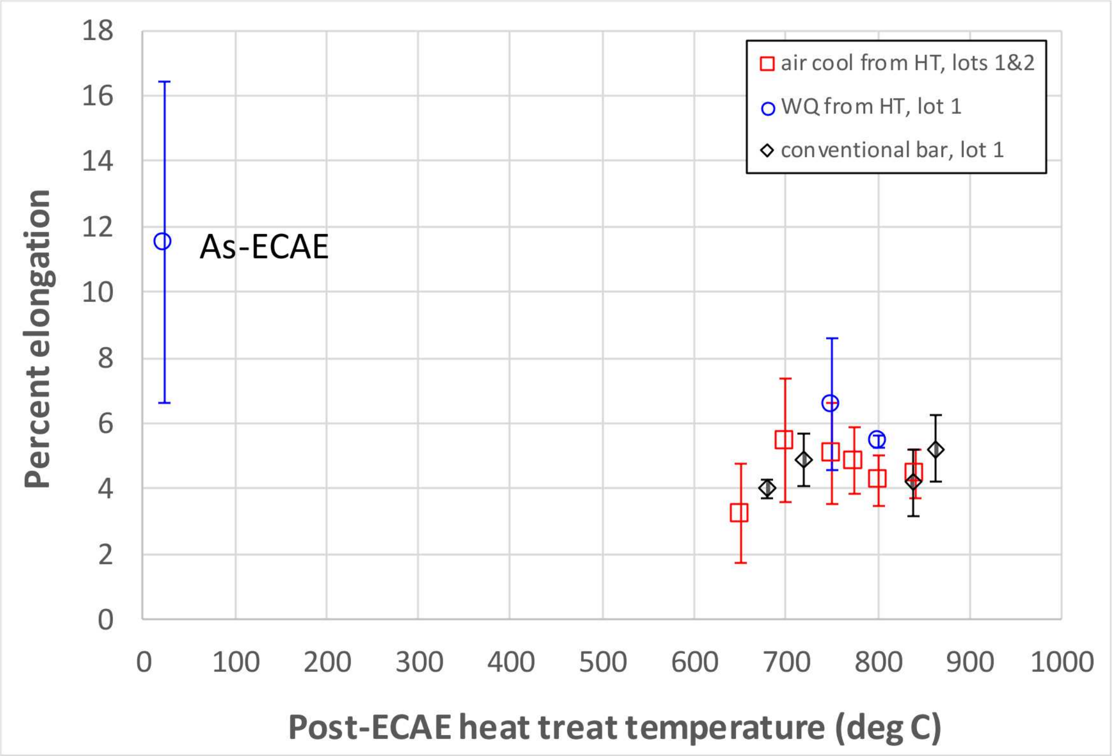
Intermediate temps. (750-800°C) may offer relatively high strength, if adequate magnetic performance can be maintained.

Testing continues on 2-pass ECAE material

Strength decreases with annealing temperature.

Effect of Heat Treatment on Ductility

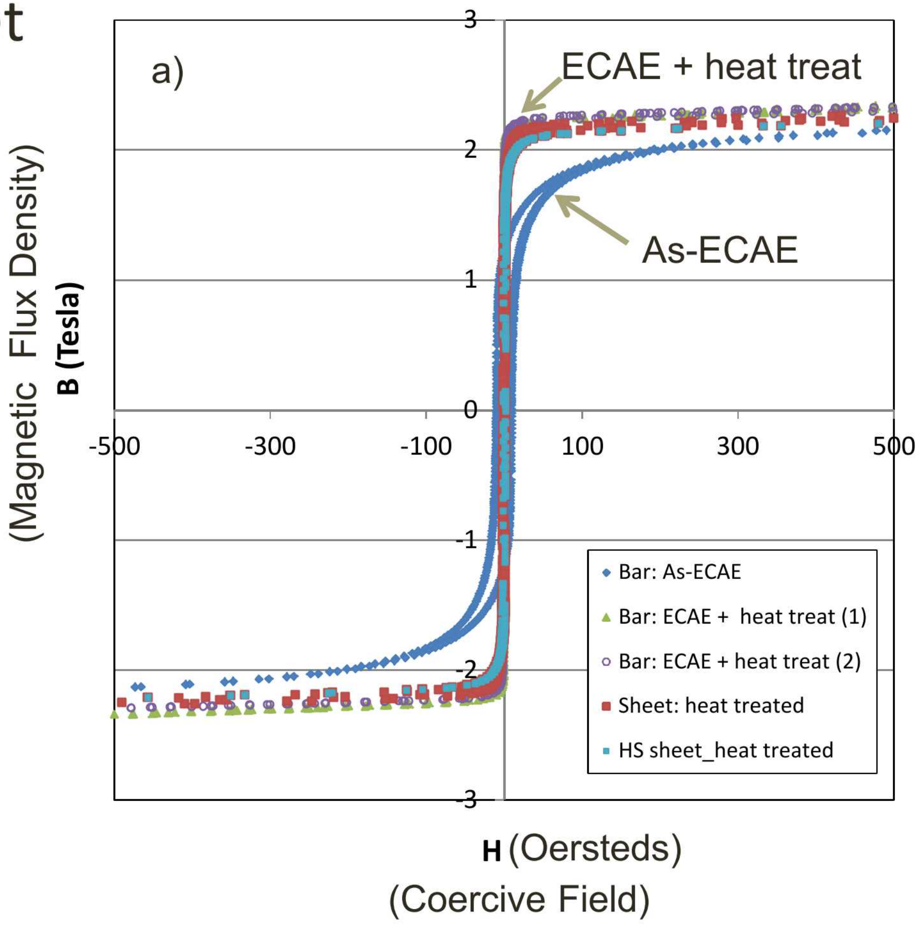
- As-ECAE material has very good ductility, likely due to its highly disordered state (bcc structure, not ordered B2).
- With the exception of 650C, post-ECAE heat treated material is no different than conventional Hiperco.
- Water quenching (fast cool) from heat treat temperature improves ductility. We have not yet measured magnetic properties of this material.



Magnetic Performance of ECAE Material

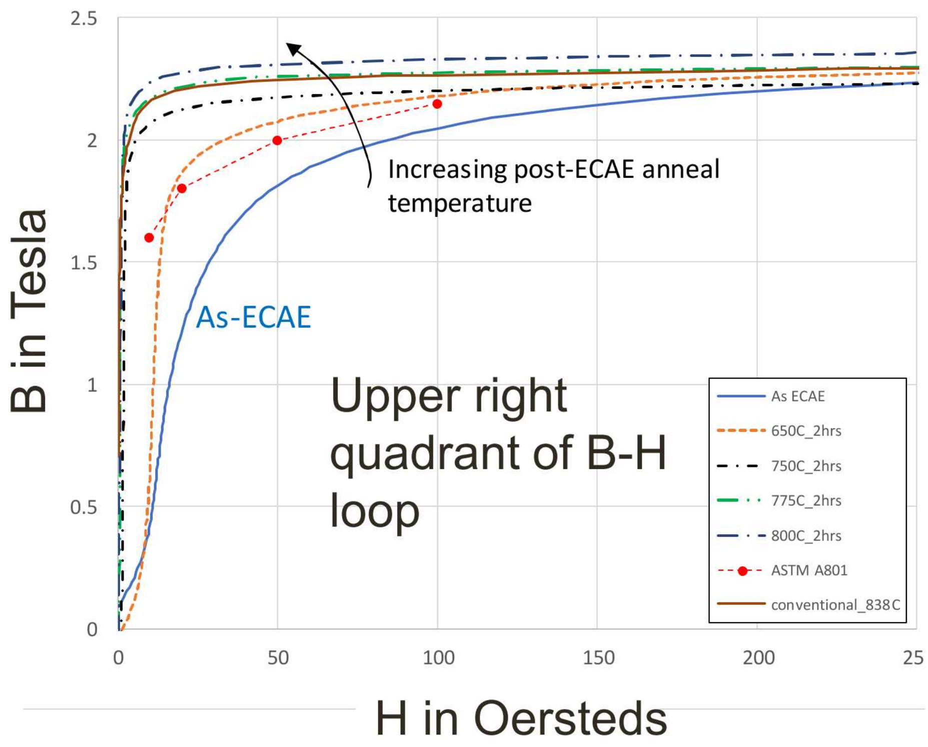
- Magnetic B-H test rings machined from ECAE material and tested according to ASTM A773
- Compared to Hiperco sheet

- 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.



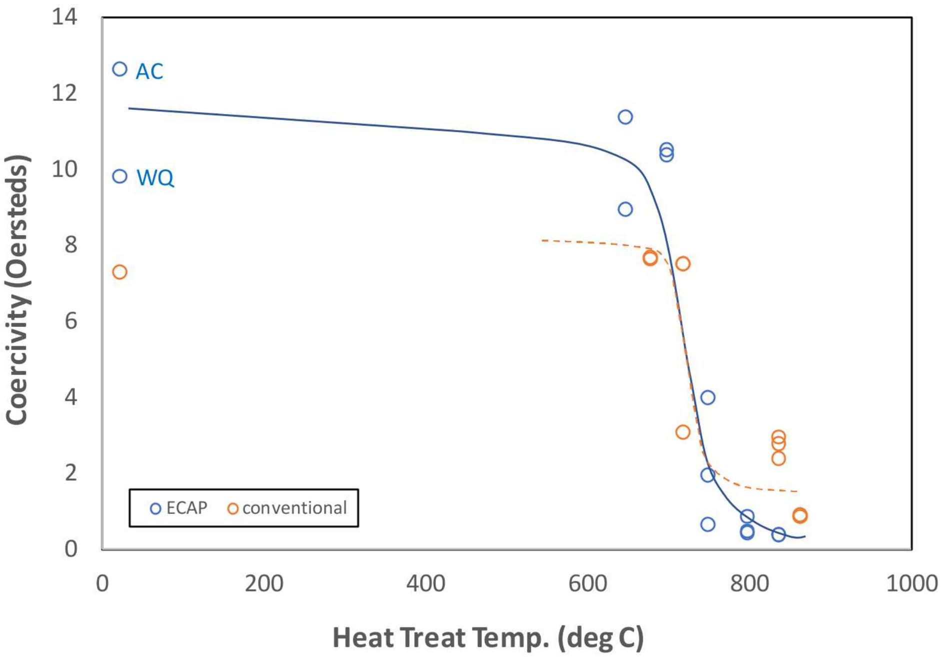
Optimization of Magnetic Behavior, Post-ECAE Heat Treat

To meet magnetic requirements, we must heat treat to at least 750°C, probably 775°C for margin.



Effect of heat treat temp. on coercivity (width of B-H loop). Unfortunately, it follows same trend as yield strength!, i.e. both controlled by annealing.

Good news: heat treated ECAE material appears to have lower coercivity than conventional Hiperco for heat treatments in the 800-865°C range.



Summary:

- ECAE is successful in the 850 to 750°C range. Significant microstructural refinement. Grain size and texture do not change with multiple passes; only one or two passes are necessary. As-ECAE condition: High strength AND high ductility.

- Full heat treatment (for magnetic properties) reduces strength and ductility to conventional bar levels. Magnetic behavior is relatively poor in the as-ECAE condition, very good in the heat treated condition. Recent optimization experiments point to 750~775°C heat treatment after ECAE for good strength AND magnetic properties.

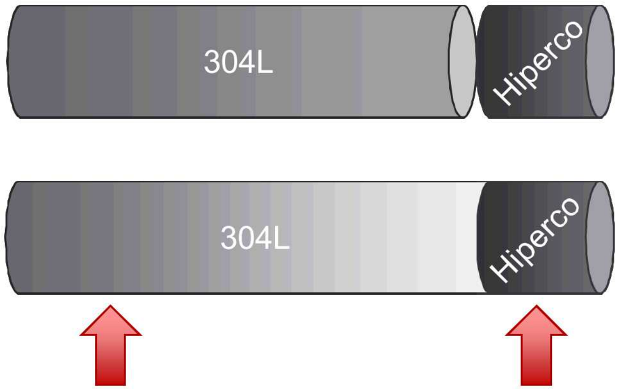
Current Work:

- Large bar sizes for ECAE being produced at a commercial vendor, Shear-Form Inc. with single pass and 2-pass ECAE. Thick stainless steel cladding prevents surface cracking observed previously. Continue to generate data on post-ECAE heat treated material. “Tech Maturation” proposal submitted.

Solid State Welding of Hiperco

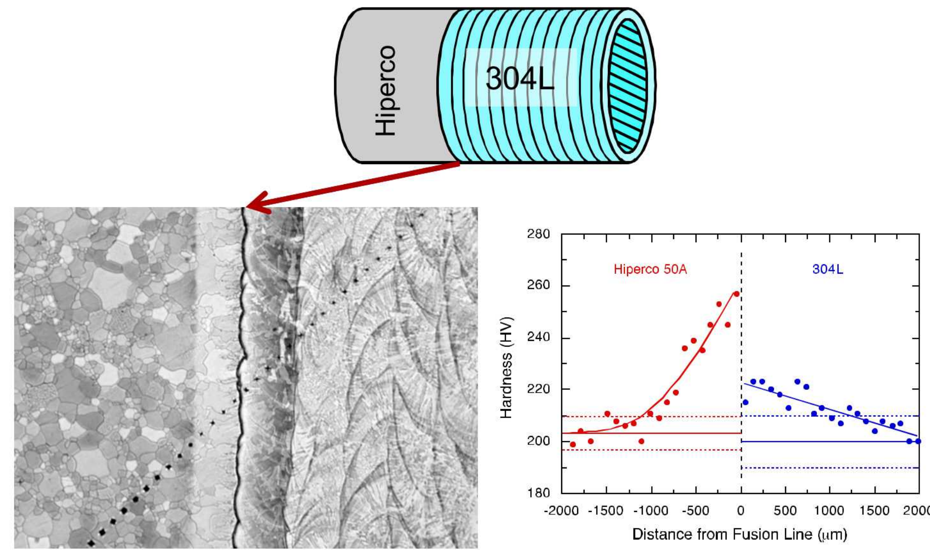
Motivation and Approach

- Evaluate <u>solid state</u> welding technique to join Hiperco to non-magnetic structural alloys to enable new design possibilities in severe mechanical environments
- Hot ductility measurements show >75% RA for T> 600° C
- Friction welding will be explored to create dissimilar solid state welds for 304L stainless steel to Hiperco 50A
  - Weld microstructure, post-weld heat treatments response, and local mechanical response will be characterized



- Highly damage tolerant
- Weldable via fusion welding processes

- Magnetic behavior only where needed

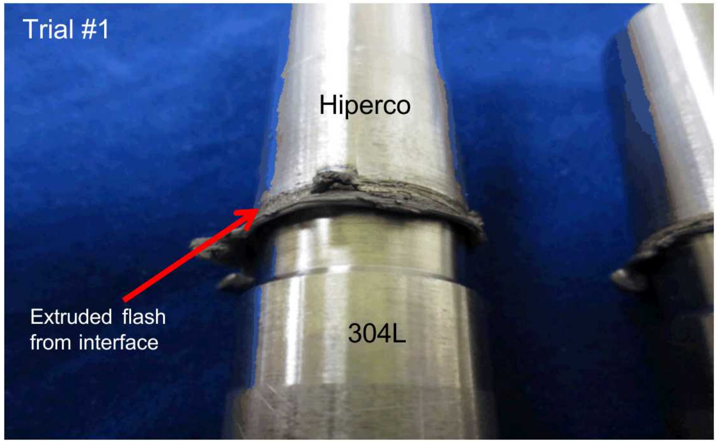


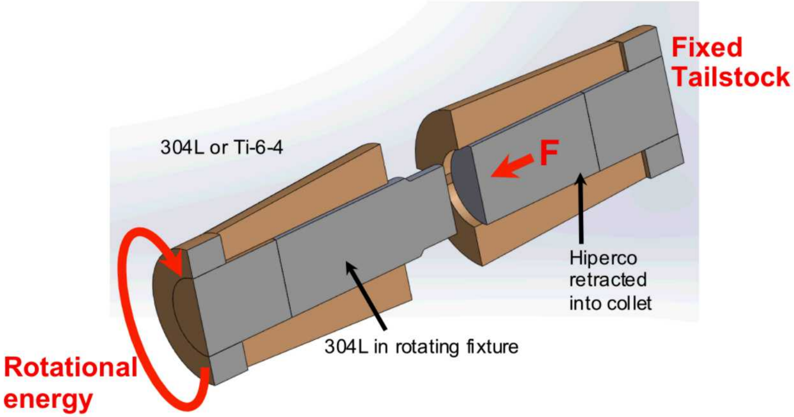
- Puskar et al. demonstrated directed energy deposition can be used to build 304L onto Hiperco
- Low ductility (0.1-1.3 %RA) was measured for dissimilar mechanical test samples

Solid State Joining via Friction Inertia Welding

- Solid state metallurgical bond created by elevated-temperature deformation resulting from frictional heating of impinging workpieces

(video)



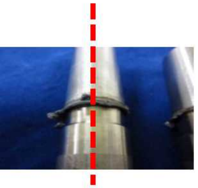


Schematic of inertia weld setup

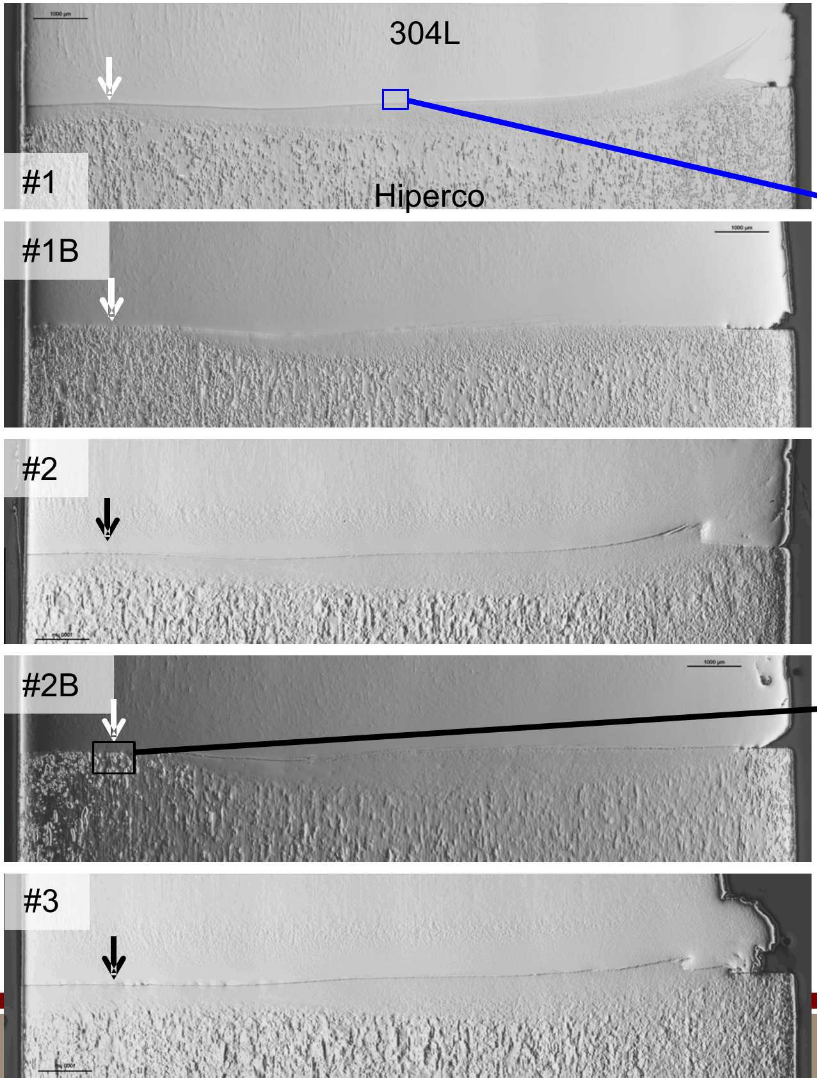
304L/Hiperco Inertia Weld Trials

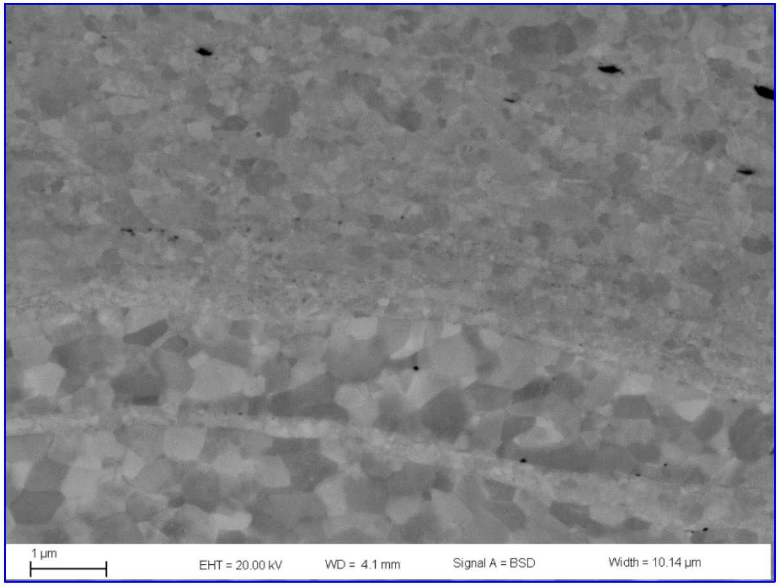
Sandia National Laboratories

- Weld at original interface shows sub-micron dynamically recrystallized grains



Transverse cross section of inertia weld sample



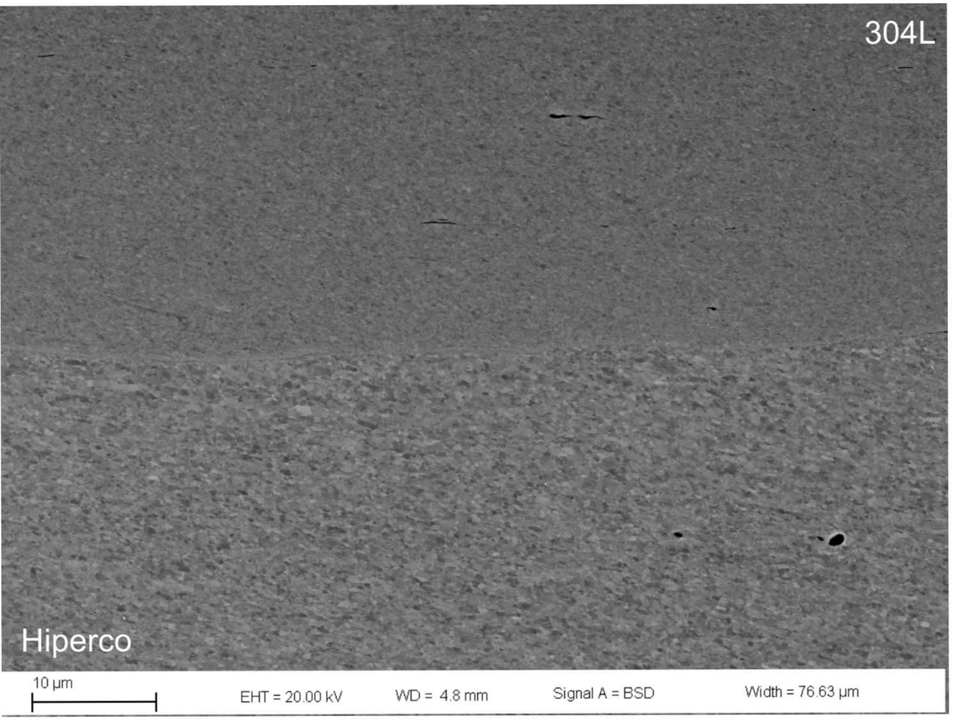


Two-Stage Welds Exhibit Improved Material Mixing

- Additional heating prior to final upset in two-stage inertia welds results in more tortuous weld interface

Single Stage

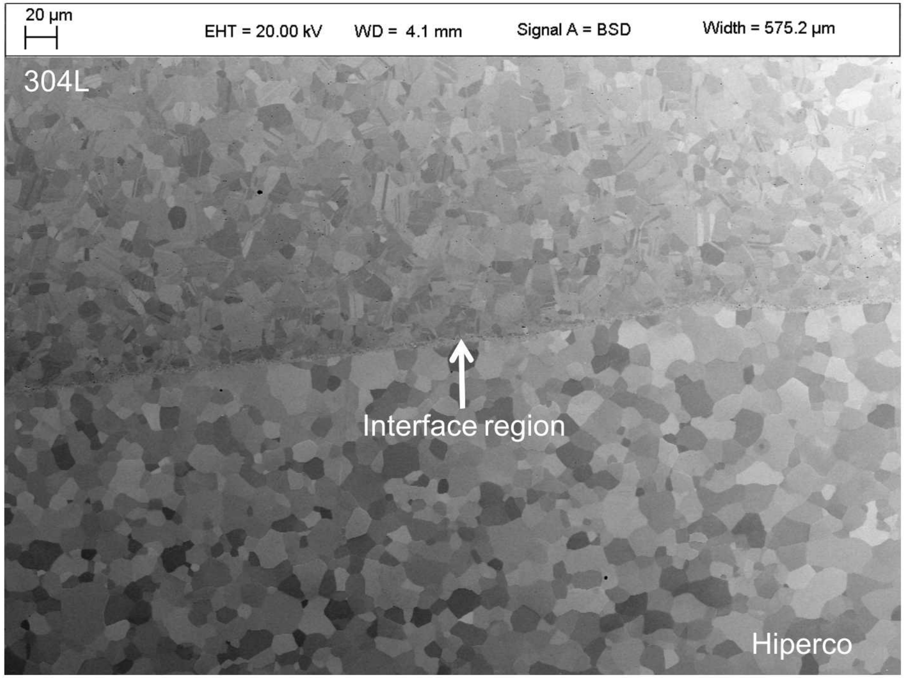
Two Stage



Magnetic Heat Treatment Results in Weld Zone Coarsening

- Post-weld heat treatment to restore magnetic properties for Hiperco results in grain growth within weld region
  - No abnormal grain growth observed

- After heat magnetic heat treatment, a narrow (~2-3 µm wide) extremely fine-grained interfacial region persists

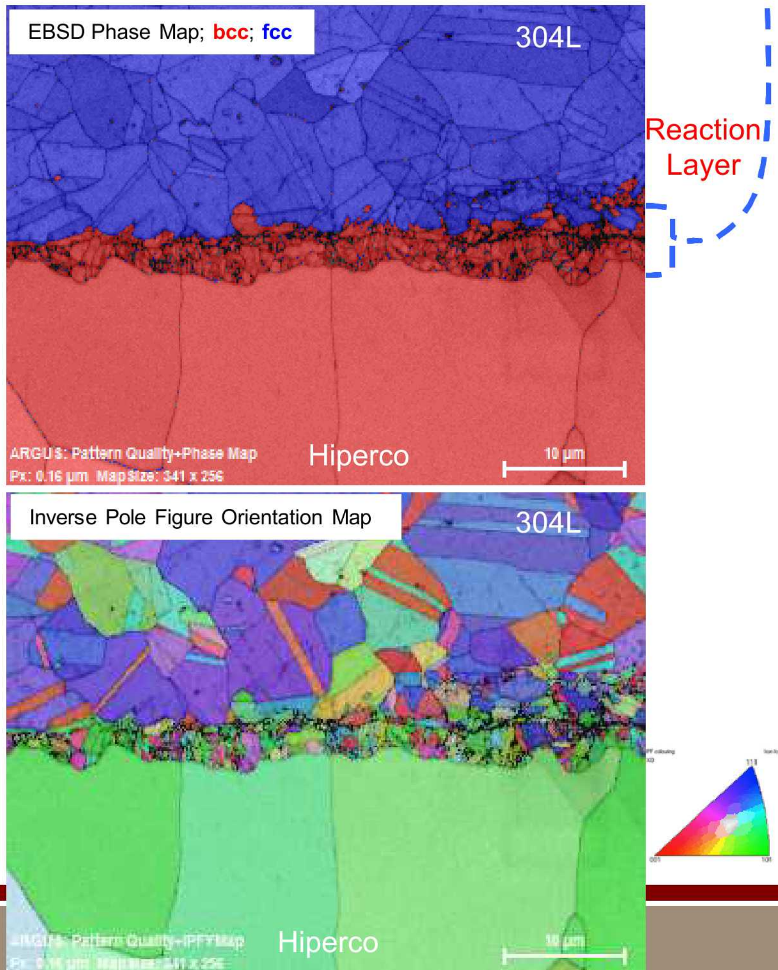


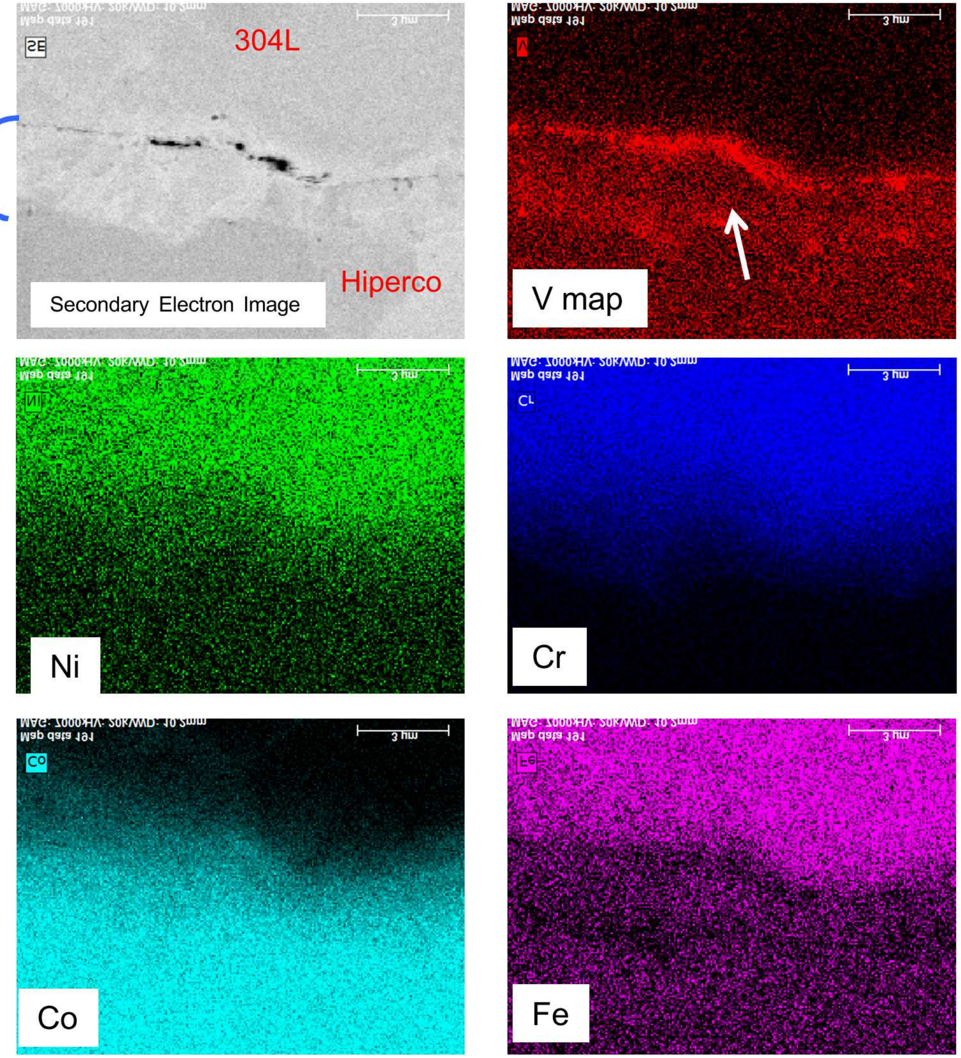
As-welded: 2-stage, #1B

Magnetic HT: 838ºC, 2hr.; #1B

Vanadium Enrichment Within Reaction Layer

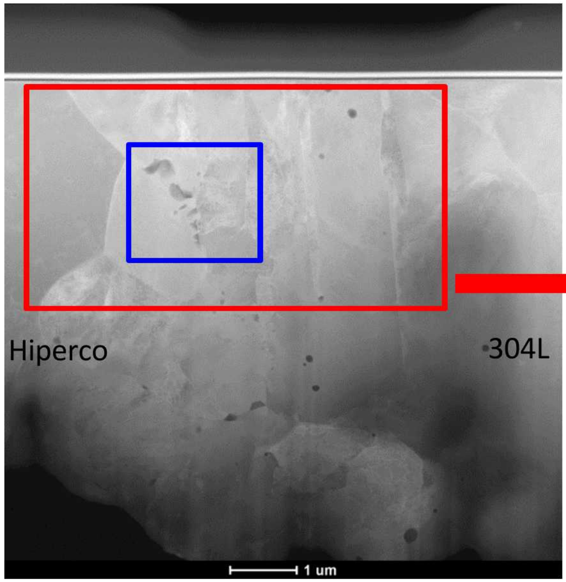
- Electron backscatter diffraction indicates fine-grained region has bcc crystal structure and is preferentially rich in Vanadium
- EDS shows evidence of interdiffusion within reaction layer

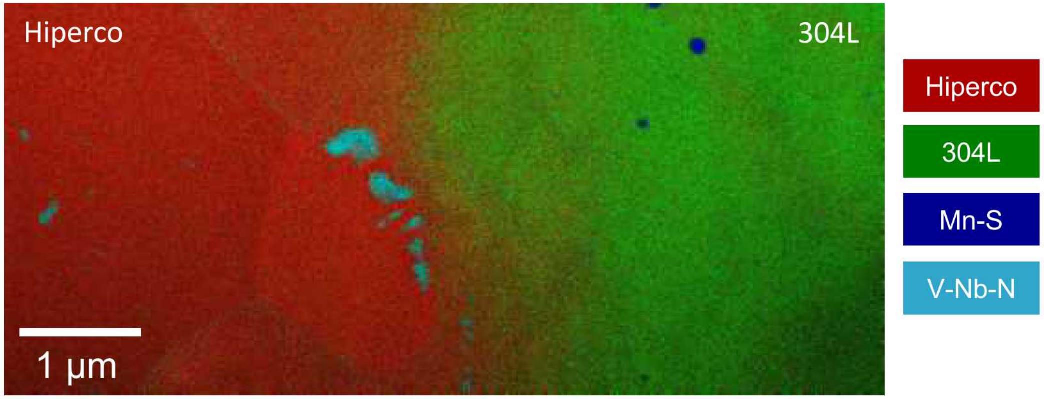


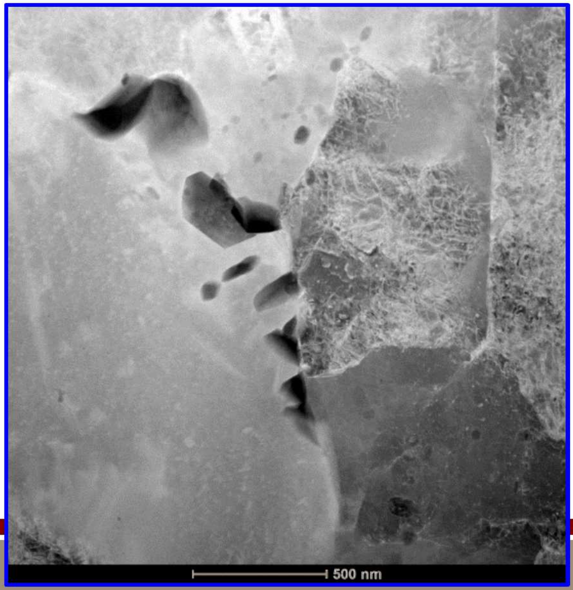


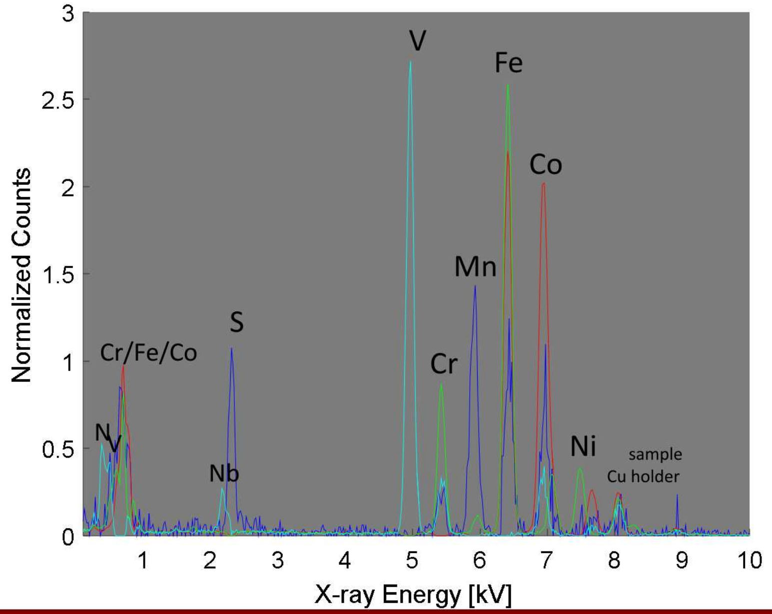
Reaction Layer Shows V-rich Nitrides after Heat Treatment

- High resolution TEM-EDS shows sub-micron particles are V-rich (V,Nb)N
- Approximate composition: 45V-50N-5Nb (at%)



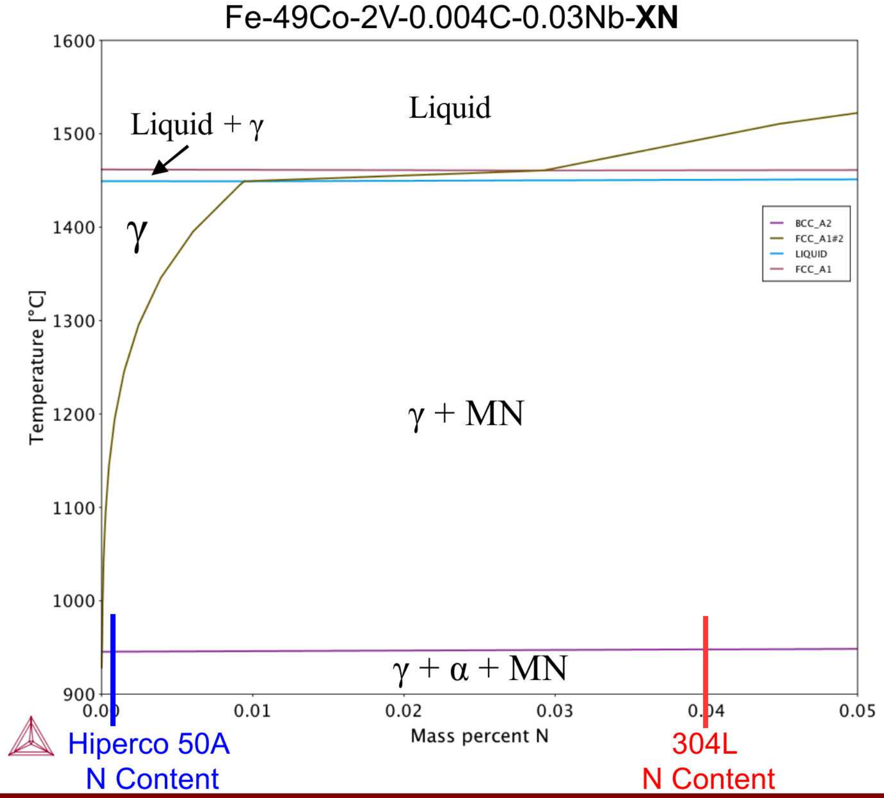


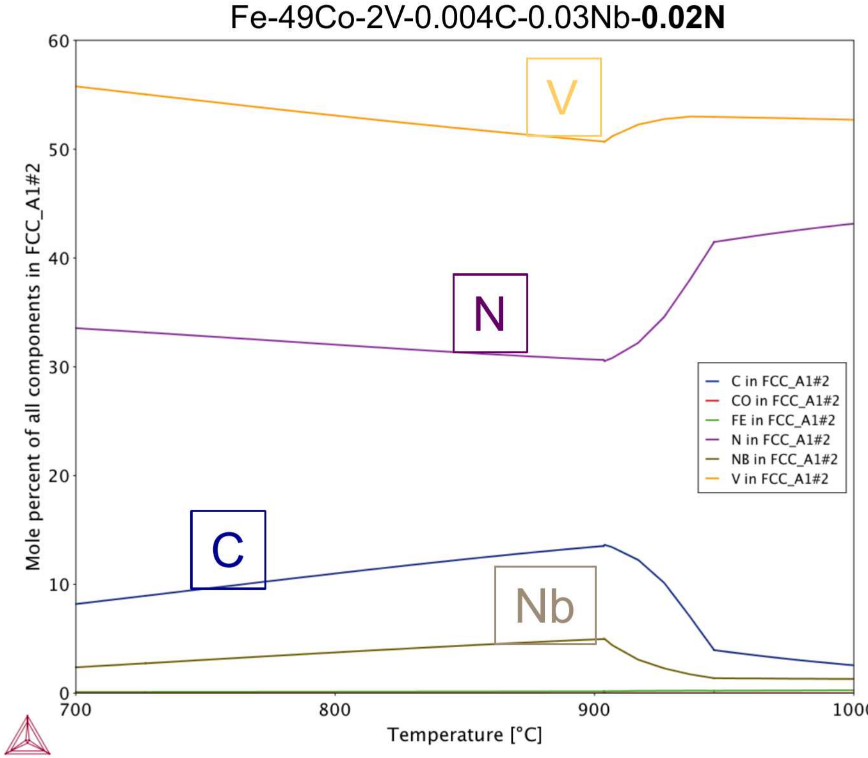




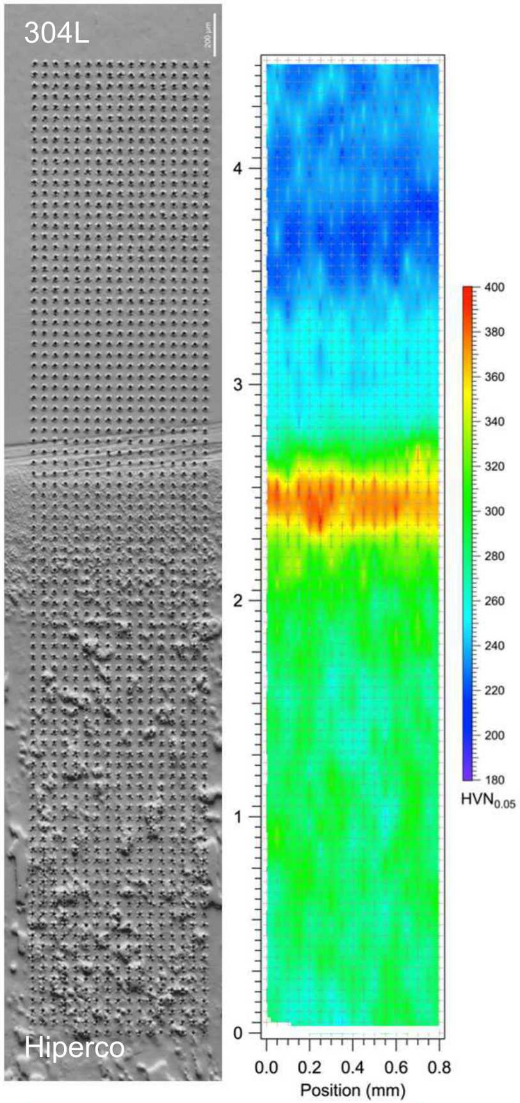
Vanadium Nitride Formation in Hiperco

- Hiperco 50A used in this study contained very low N levels
  - 10 wt. ppm by inert gas fusion
- Multicomponent thermodynamic prediction indicate indicates nitride phase stoichiometry consistent with TEM observations

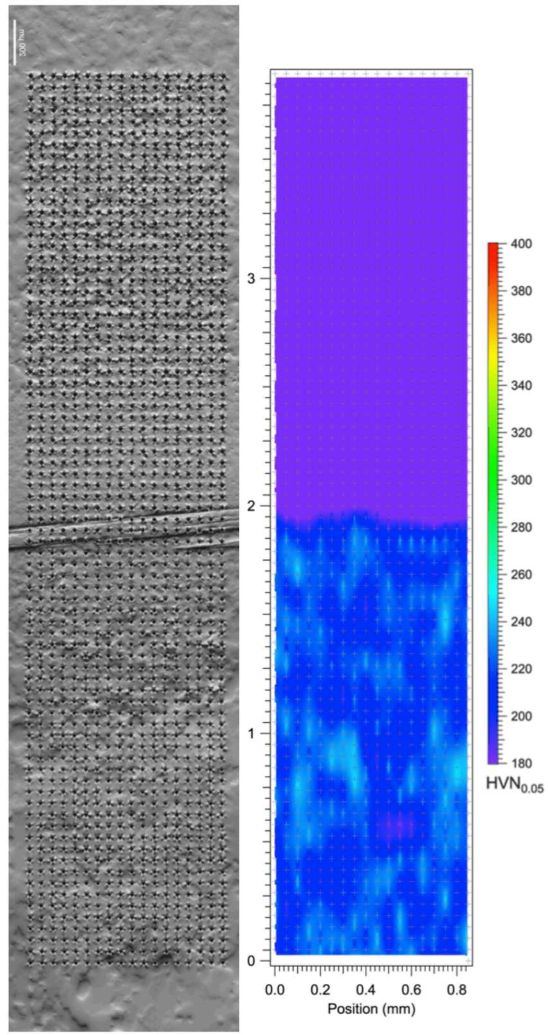




Microhardness Mapping Used to Assess Weld Zone Mechanical Properties



As-welded: 2-stage, #1B

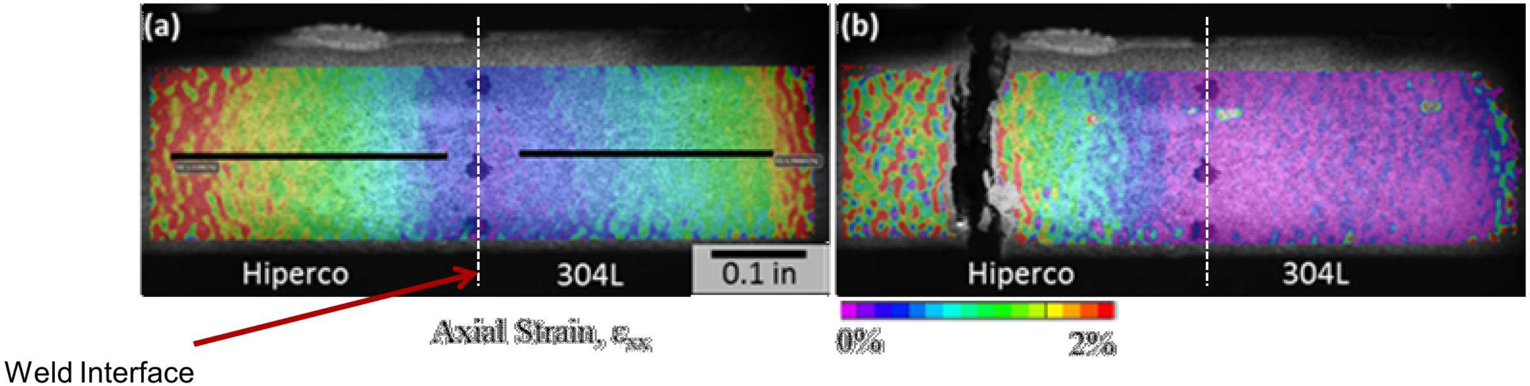


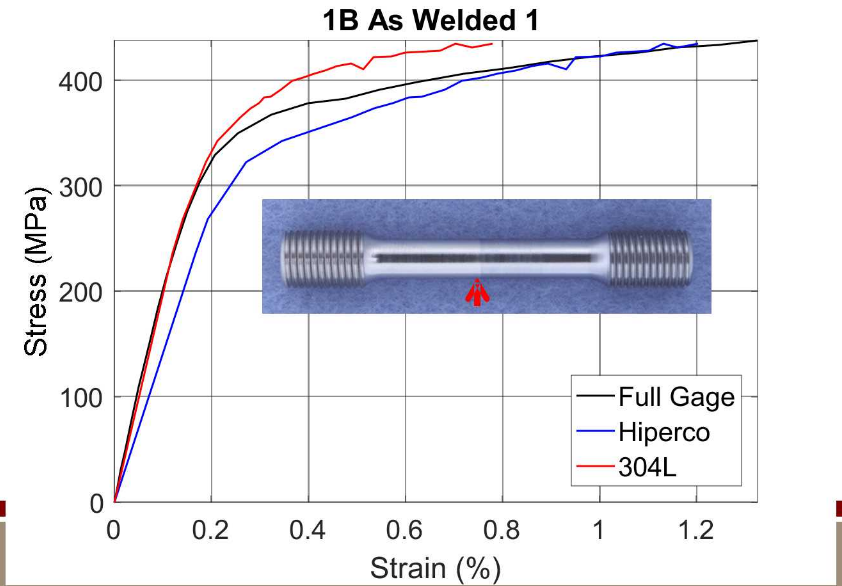
Magnetic HT: 838ºC, 2hr.; #1B

- Fine dynamically recrystallized grains in weld zone as-welded result in apparent strengthening near the original interface
  - ~200 HV gradient as-welded
- Static recrystallization/grain growth operative during post-weld heat treatment reduces gradient in hardness between 304L/Hiperco
  - ~60 HV gradient
- Any effects from thin (2-3 µm) reaction layer were missed by microhardness indents

Mechanical Testing of As-Welded 304L/Hiperco Reveals Brittle Failure in Hiperco away from Weld

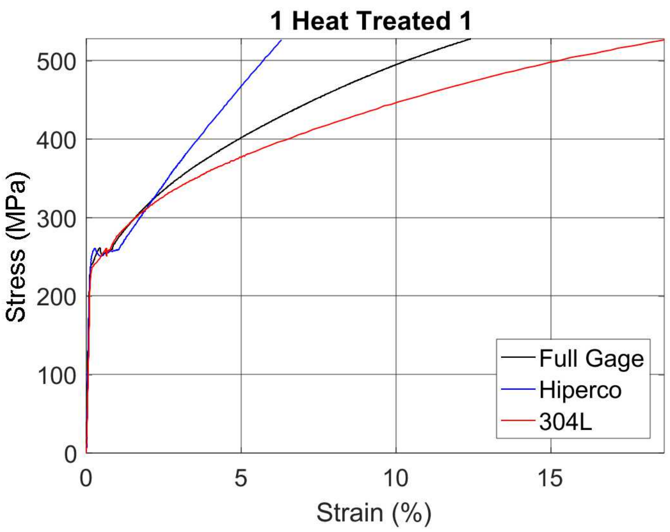
- Strain concentrates in Hiperco away from weld zone
  - Hiperco without magnetic heat treatment (i.e., disordered) has poor ductility (1-3% strain to failure)
- Fracture occurs in Hiperco remote from weld zone

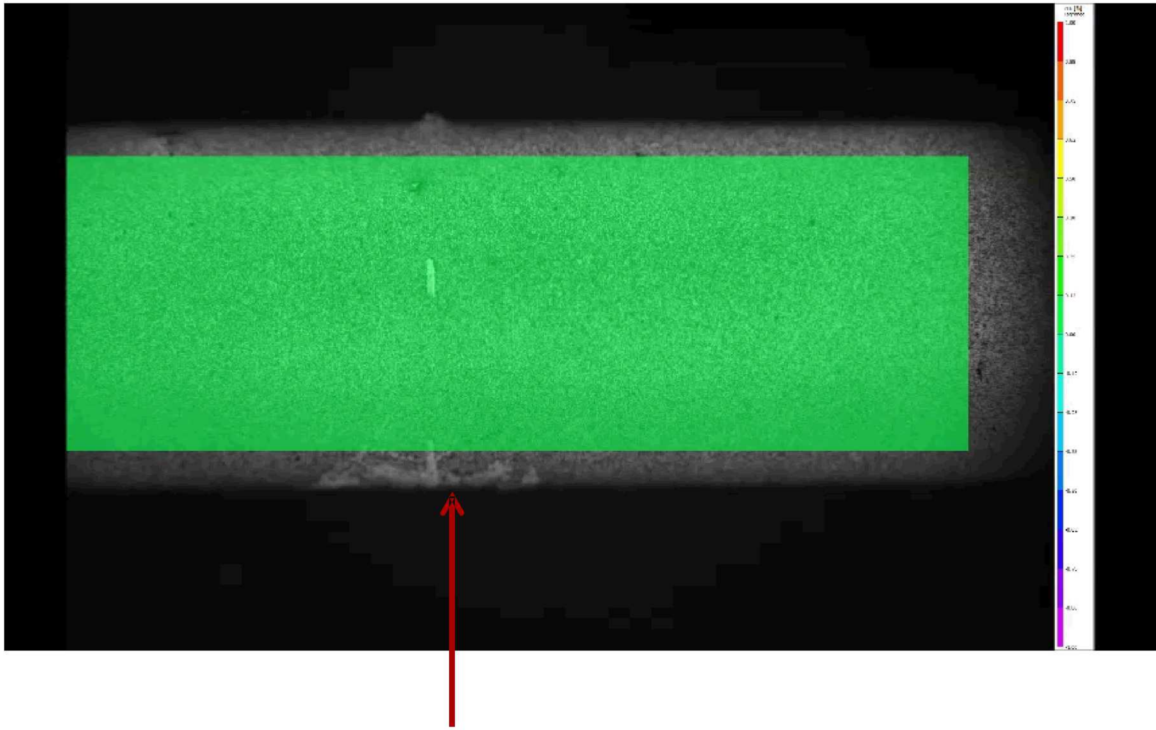




Post-Weld Heat Treatment Results in Improved Inertia Weld Mechanical Behavior

- Despite ordering reaction in Hiperco resulting from PWHT, advantageous mechanical behavior observed—13% global strain to failure.
- DIC movie shows plastic strain transfer from Hiperco to 304L resulting in overall increase in strain-to-failure relative to Hiperco alone

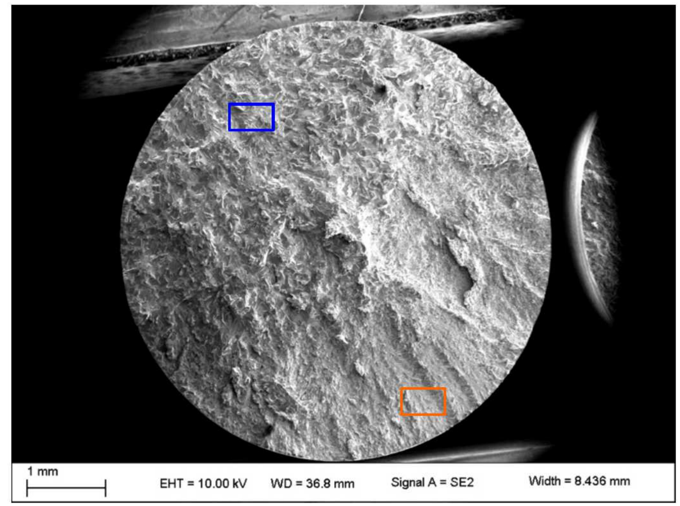


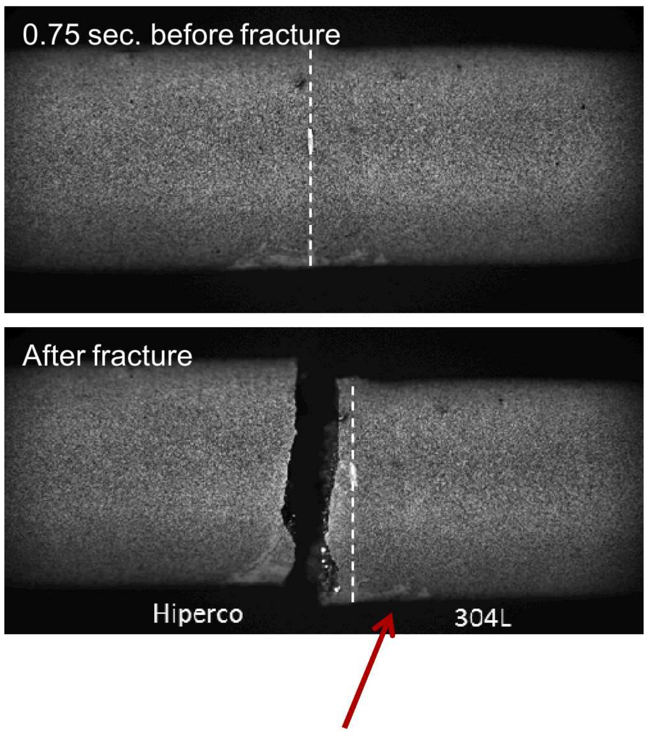


Weld Interface

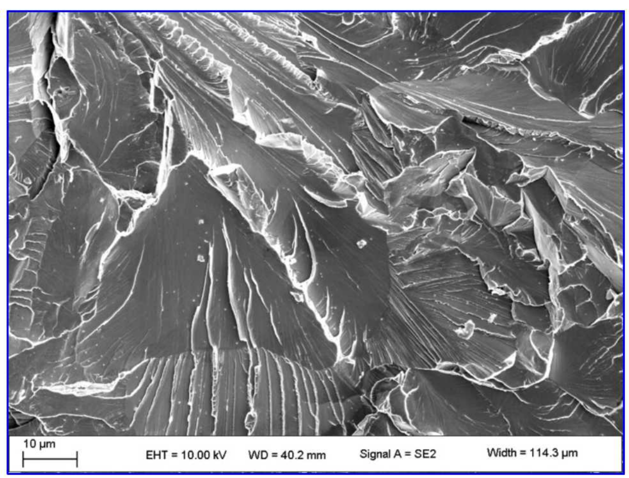
Fractographic Analysis

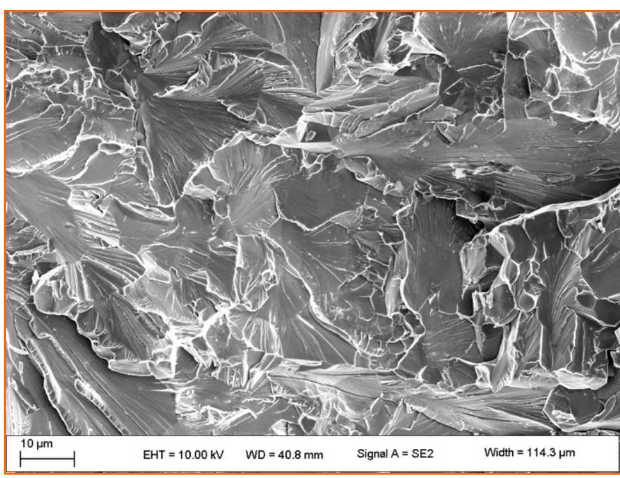
- Despite macroscopic ductile behavior measured via DIC, final fracture in Hiperco occurred via brittle transgranular cleavage





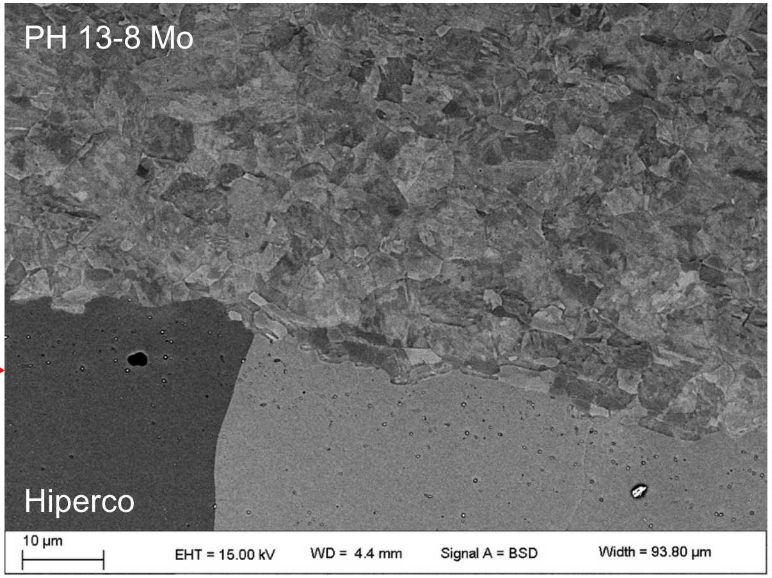
Cross sectional area reduction in 304L



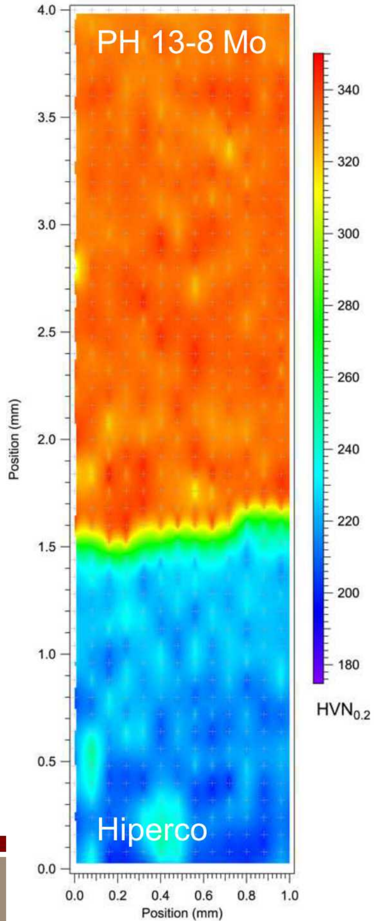


Other Dissimilar Alloy Combinations: PH13-8 Mo/Hiperco

- Interface characteristics similar to Hiperco/304L
- Advantageous mechanical behavior exhibited by not likely with PH13-8 Mo due to strength discrepancy– DIC tensile testing underway
- Hiperco magnetic heat treatment not optimized for PH13-8 Mo strength

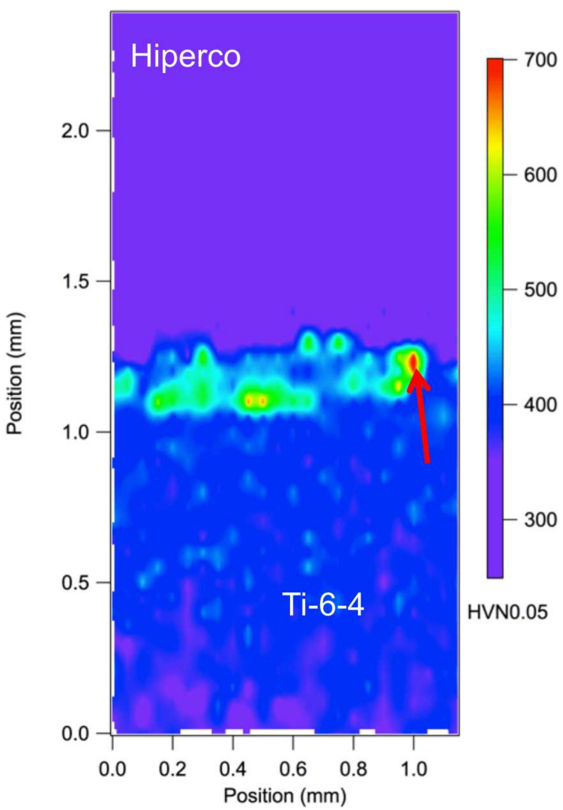


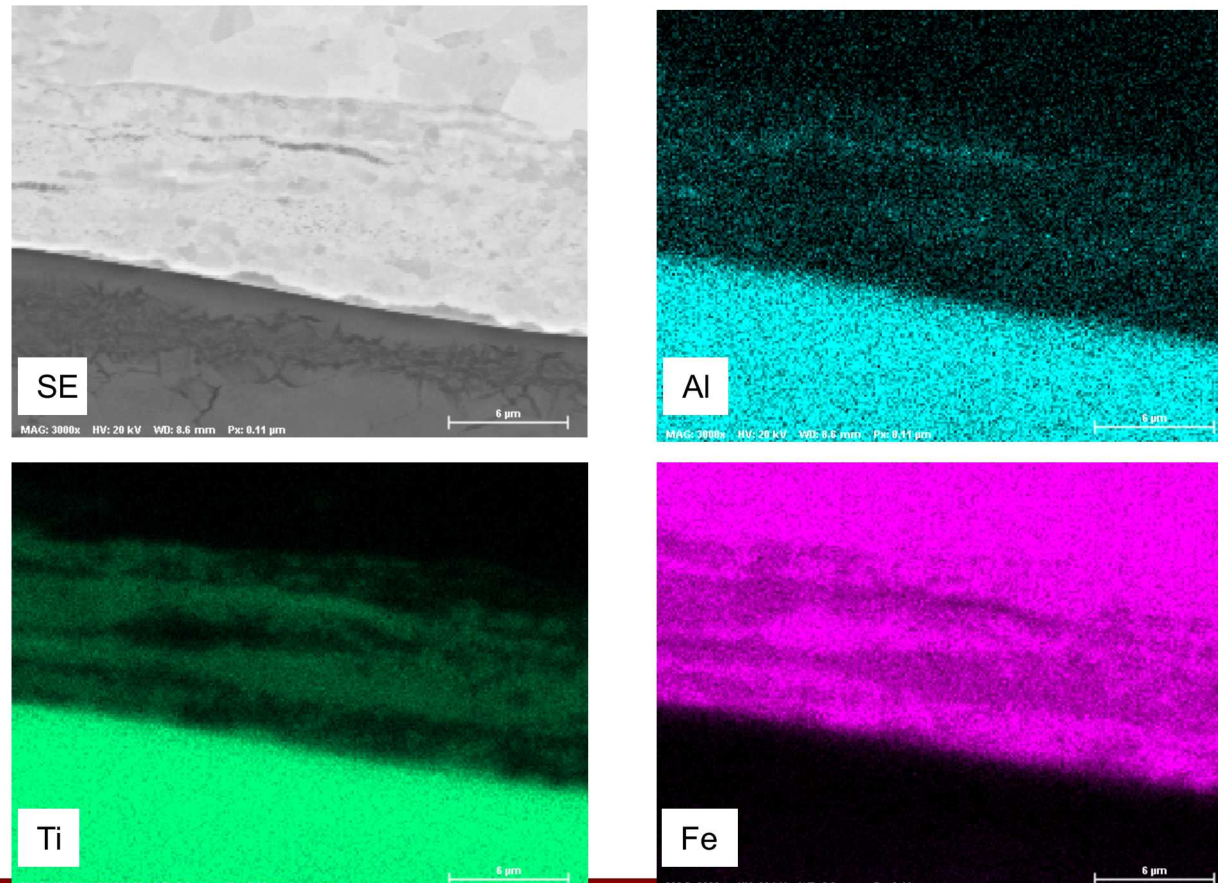
Heat Treated (838°C)



Other Dissimilar Alloy Combinations: Ti-6Al-4V/Hiperco

- High-hardness, brittle intermetallic phases observed in as-welded condition





Conclusions

- Friction inertia welding demonstrated as robust method for dissimilar welding of 304L to Fe-Co-V (Hiperco)
- Two-stage weld cycle results in improved interfacial material mixing
- Post-weld heat treatment to restore Hiperco magnetic properties coarsens grains, reduces mechanical property gradient, and results in advantageous composite mechanical behavior
- Improved strain-to-failure of 304L/Hiperco bi-material samples can improve performance of electromagnetic assemblies in severe mechanical environments
  - Inertia welded 304L/Hiperco opens new design possibilities