

Advances in AIMI “Second Generation” MgB₂ Wires, n-values, and Coils

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Funded by a the state of
Ohio, NIH, and a DOE
SBIR

M. A. Rindfleisch, M. J. Tomsic, C. J.
Thong, D. Doll, HyperTech Research



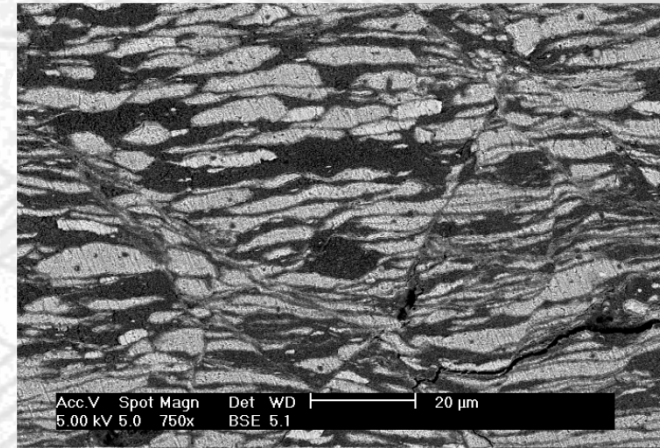
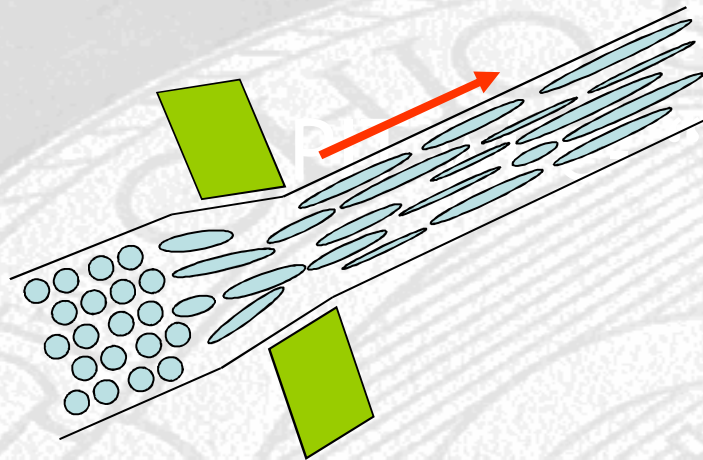
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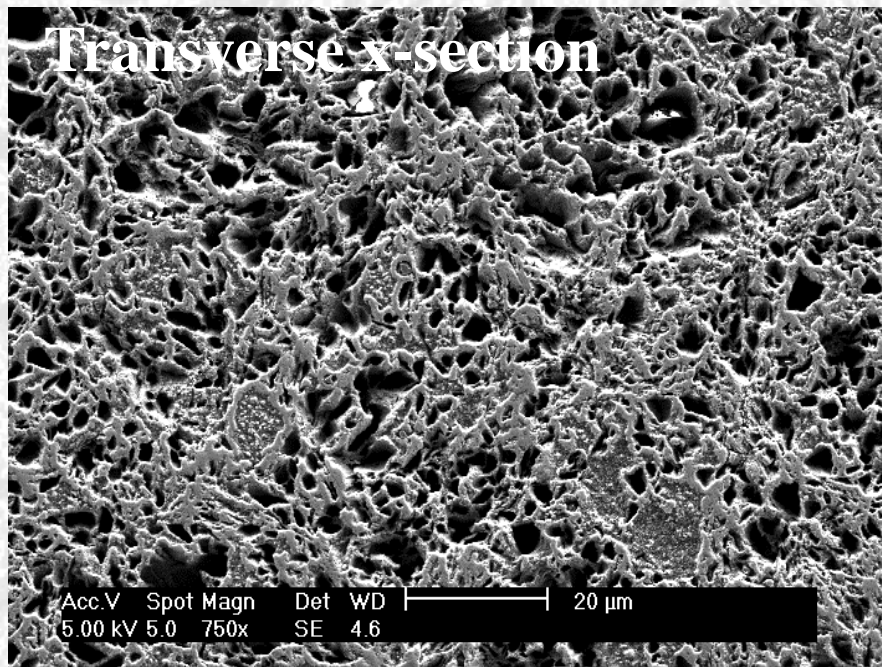
Outline

1. Transport Results for Multifilamentary Advanced Internal Magnesium Infiltration (AIMI) (2G) strands
2. Large increases in n -values for PIT strands at 4 K and as a function of B and T
3. Preliminary results for Wire-In-channel NZP and MRI-like Coil Winding

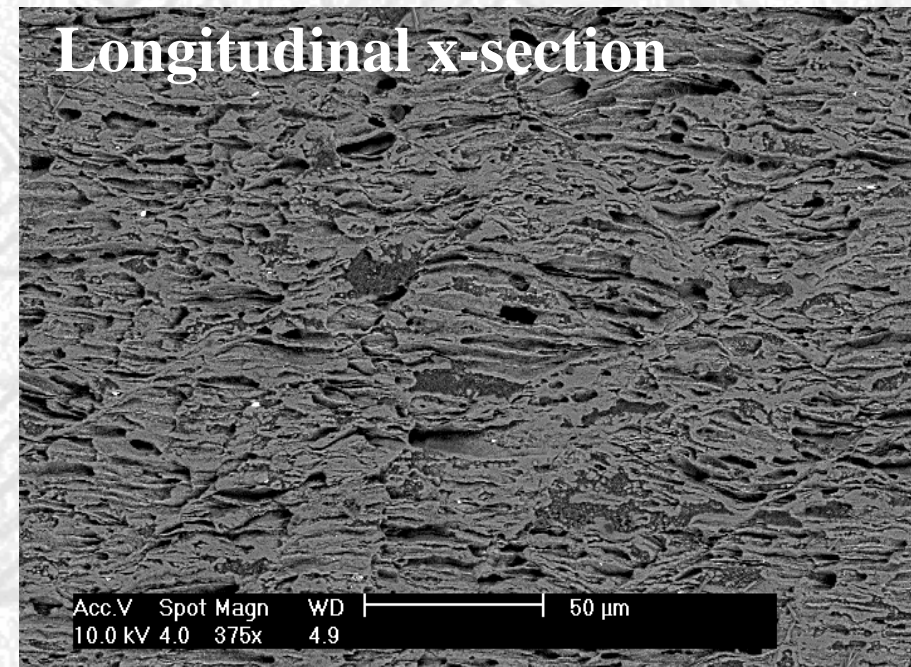
PIT Conductors have porosity



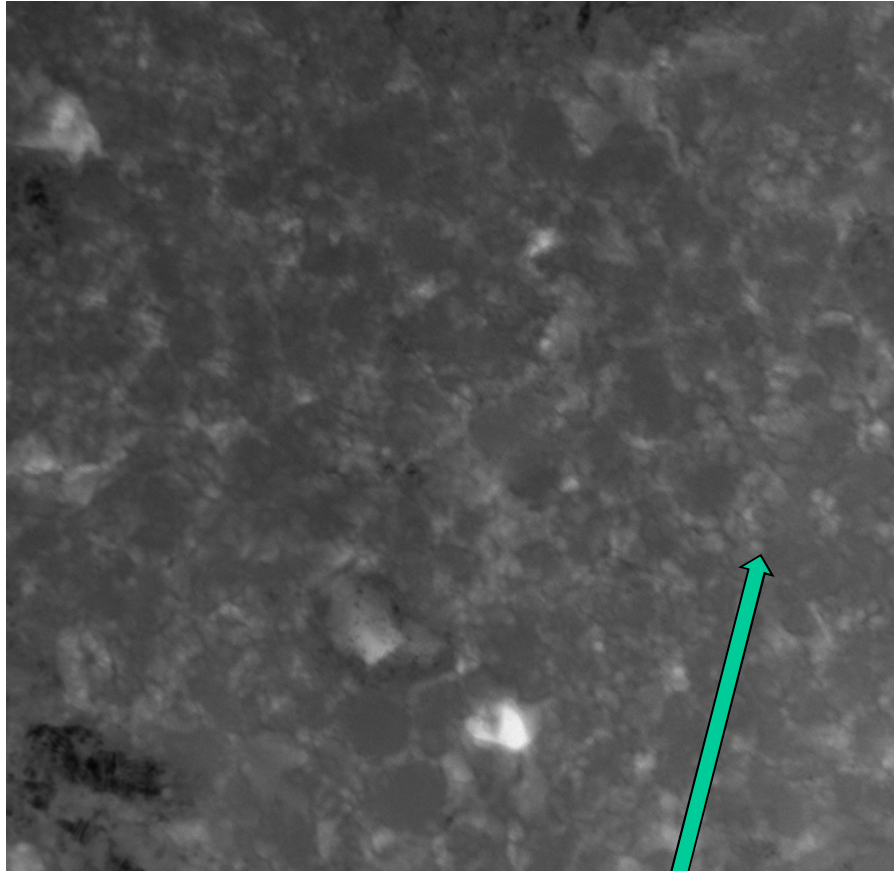
Transverse x-section



Longitudinal x-section

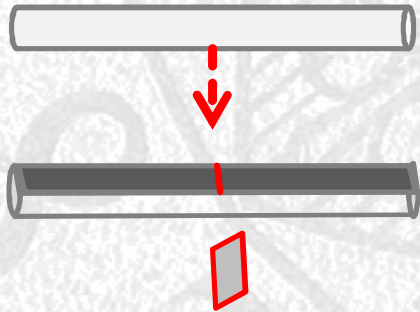


**But even in the
“solid” regions,
oxides limit current!
BF TEM MgB₂ wire
x-section**

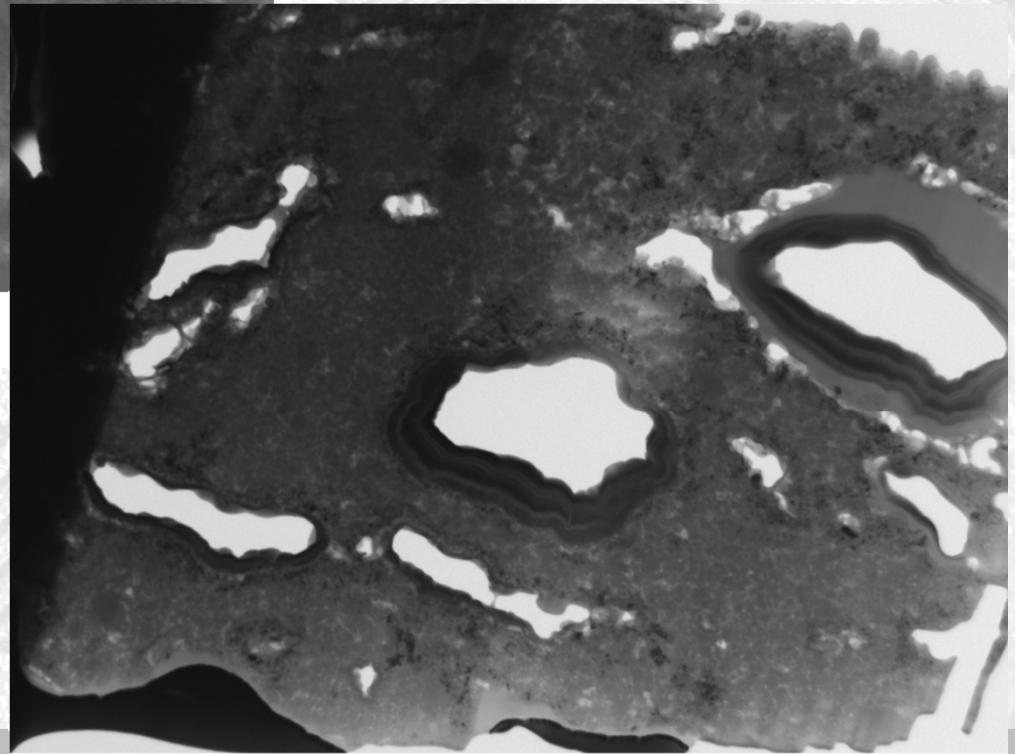


1391_012.tif
Cal: 0.985222 nm/pix

100 nm



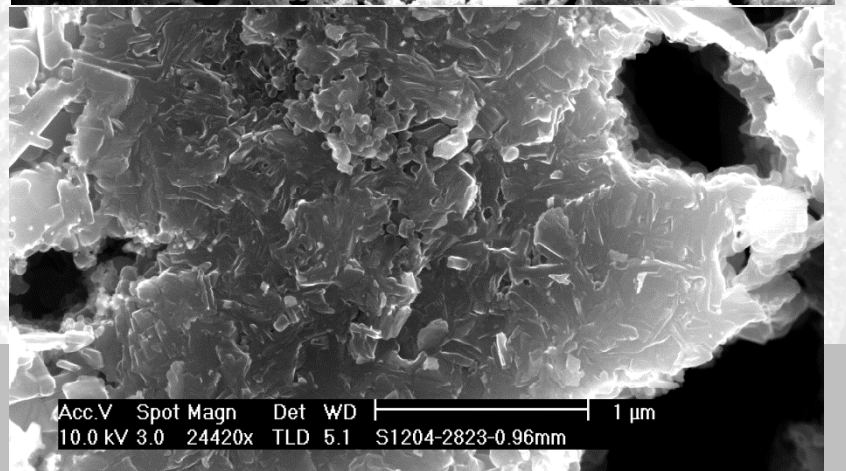
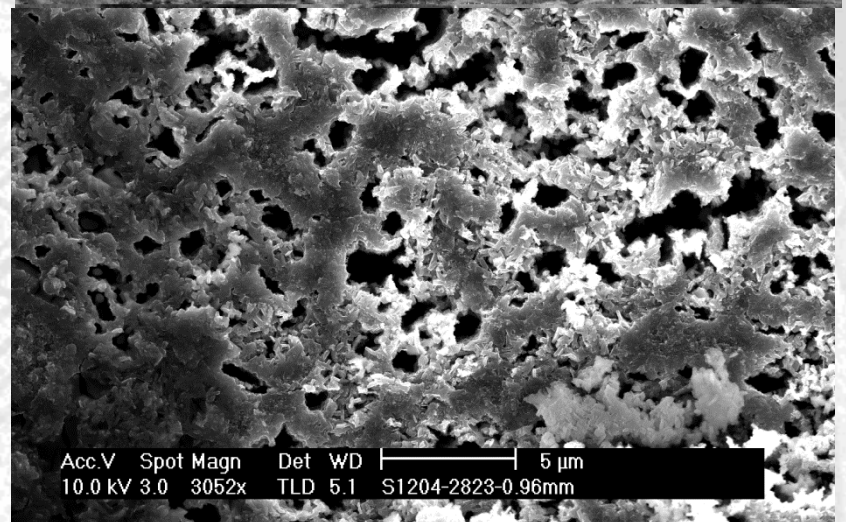
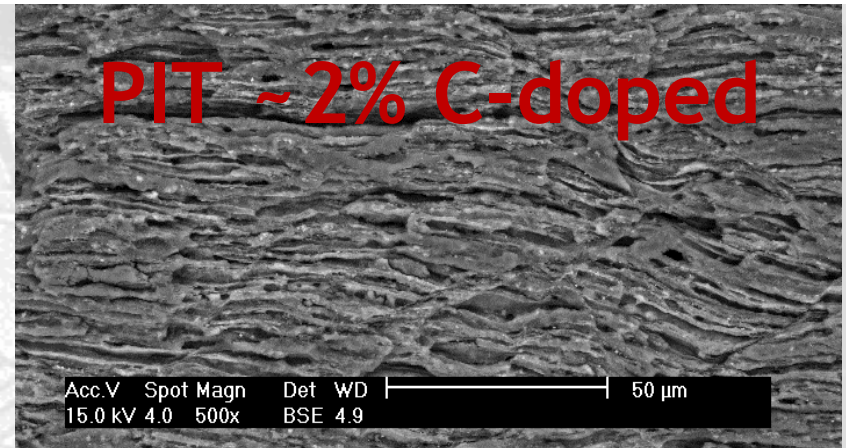
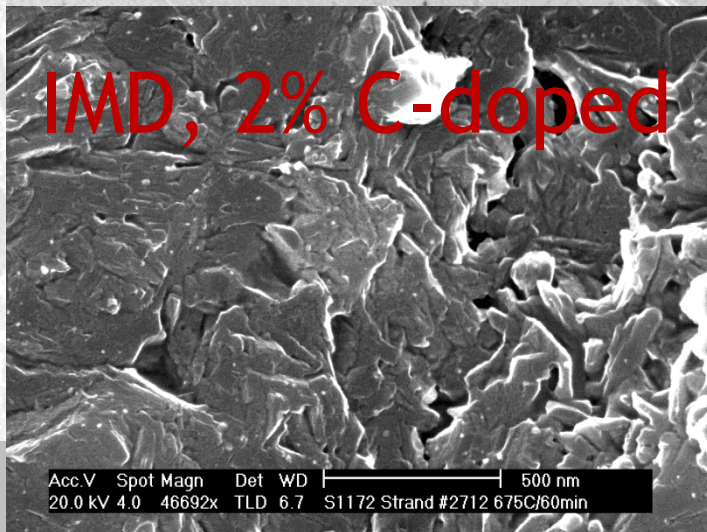
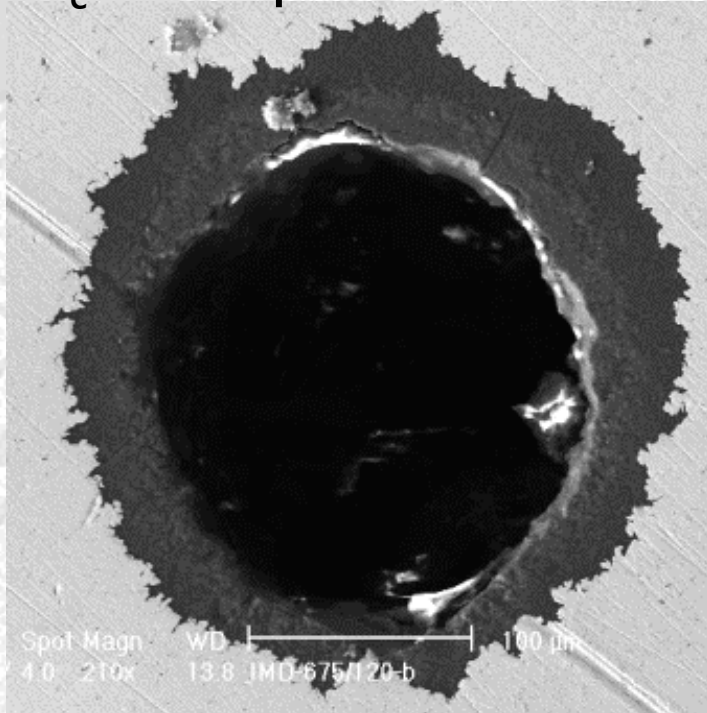
Notice
marbled
structure ---
oxides at GB



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2 μm

But we could get much higher layer J_c if we prevented this



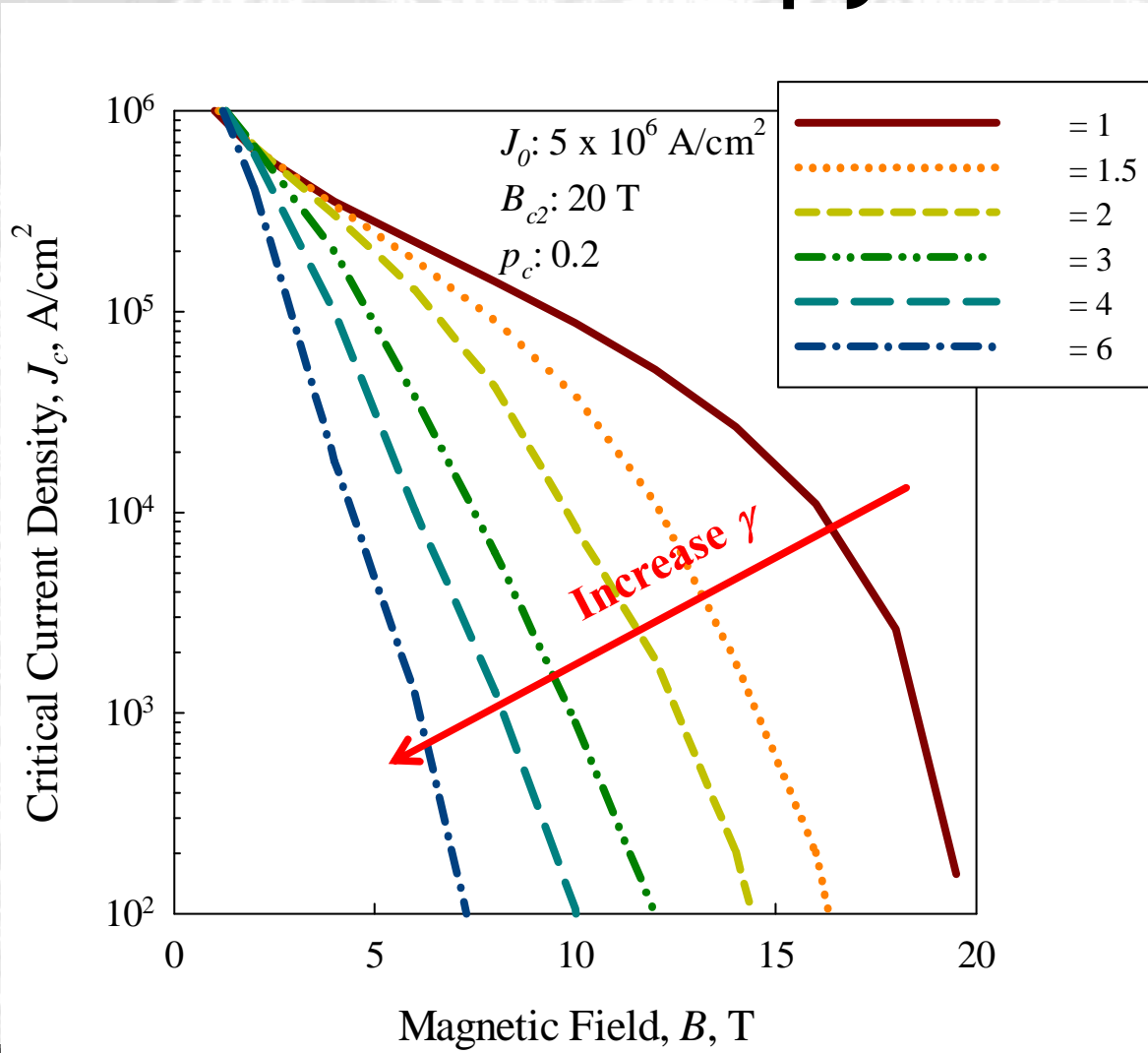
What is the effect of Anisotropy?

Use a modified Eisterer model

- J_0 : 5×10^6 A/cm²

- B_{c2} : 20 T

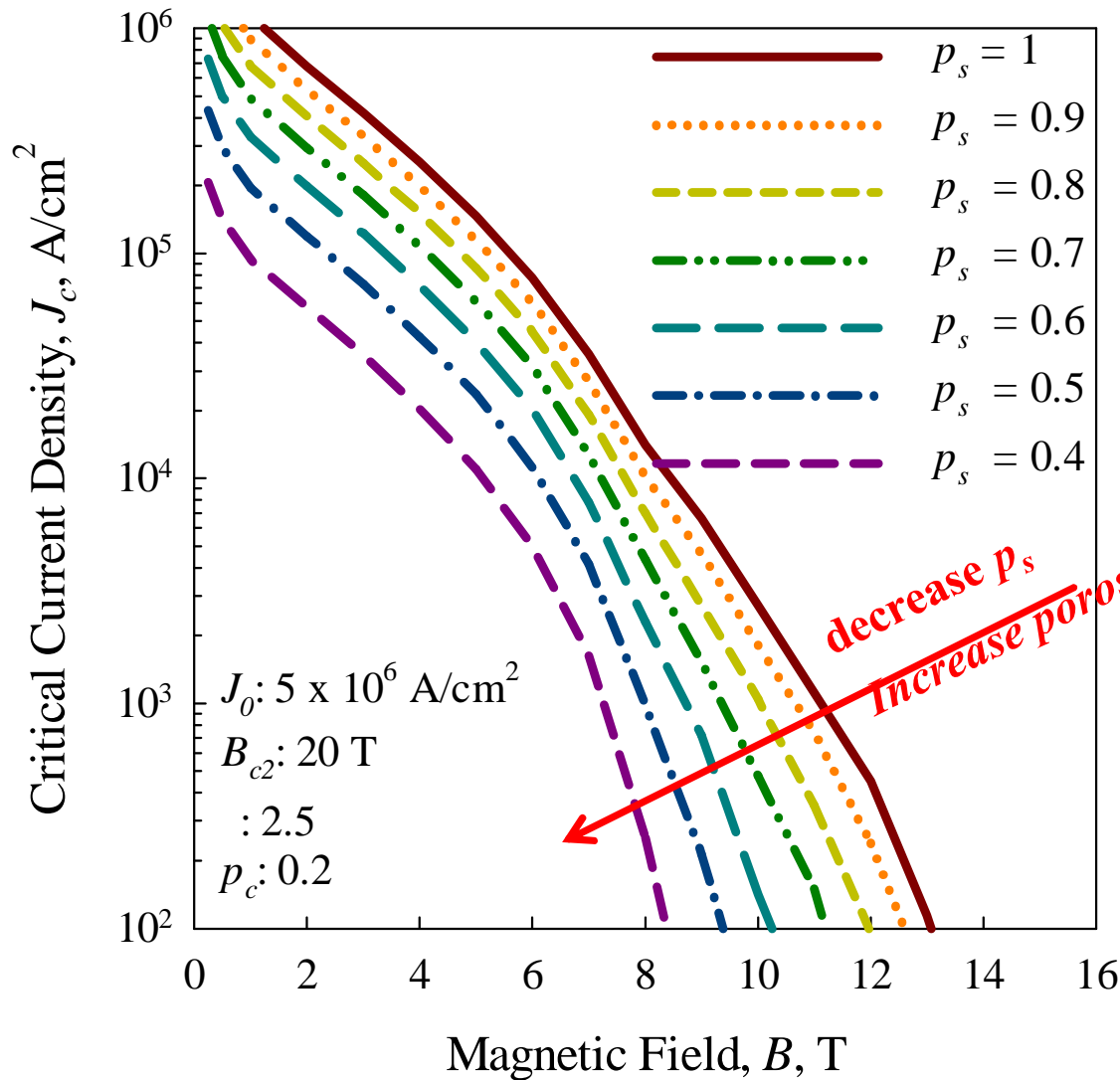
- p_c : 0.2- densely packed system



Increasing γ causes a decrease in the amount of supercurrent percolation paths

As a result, J_c at high B decreases - effect intensifies with increase in γ

How about the influence of Porosity?



Anisotropy (γ) fixed to 2.5

$p_c: 0.2$

$B_{c2}: 20 \text{ T}$

$J_0: 5 \times 10^6 \text{ A/cm}^2$

Porosity globally decreases $J_c(B)$



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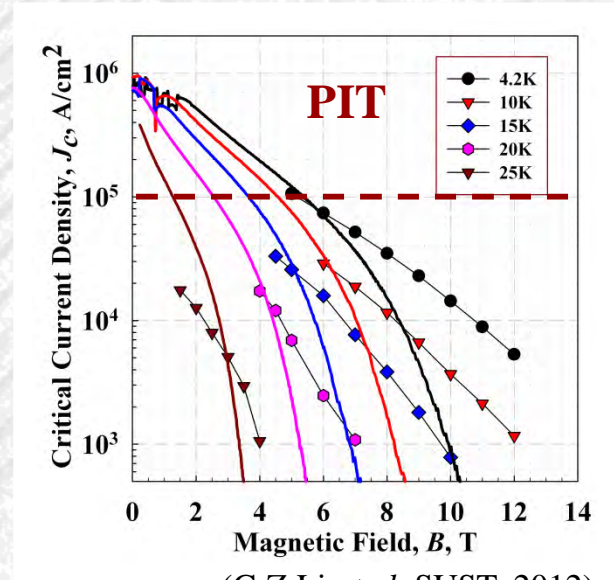
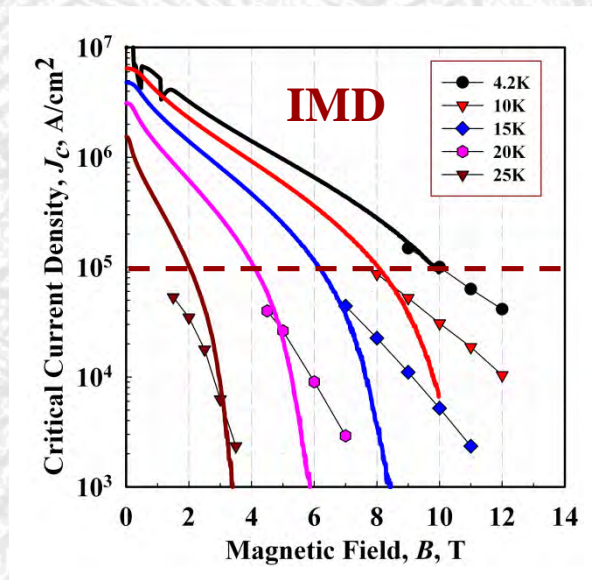
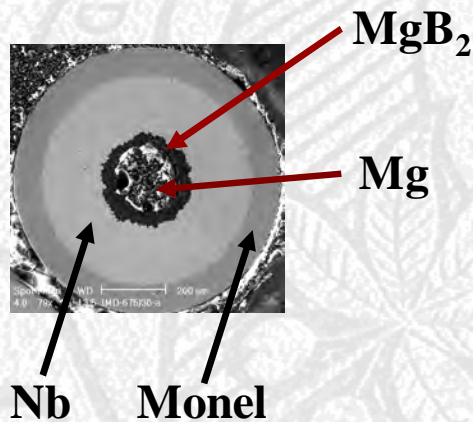
So....

- PIT MgB₂ is limited by the effects of nanoscale non-optimum connectivity, in addition to the larger scale porosity
- Thus, when MgB₂ is densified, we get a factor of 10, rather than a factor of 2
- Higher field drop off is sometimes entirely attributed to anisotropy, but about half comes from nanoscale connectivity
- Application of percolation theory to MgB₂ wires of different designs isolated the effects of porosity and crystalline anisotropy.
- Porosity was seen to be just as important a factor as
 - In particular, at 10 T, 60% of supercurrent degradation comes from anisotropy, 40% from porosity
 - Can nearly eliminate the latter, reduce the former through doping



Motivation

1. Diffusion processed wire (Mg-RLI or IMD): initiated by Giunchi *et al.* (Edison SpA) – very dense, but low J_c because of powder type
2. Developed by Kumakura *et al.* (NIMS) – their B_c , much higher J_c s
3. Further developed at OSU-HTR – use of very fine B_c , and C-doping led to even higher J_c s



(G Z Li *et al.*, SUST, 2012)

■ high layer J_c . 1.0×10^5 A/cm² at 4.2 K, 10 T



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Measurement of J_c and n for monofilaments

IOP PUBLISHING

SUPERCONDUCTOR SCIENCE AND TECHNOLOGY

Supercond. Sci. Technol. 25 (2012) 025001 (10pp)

doi:10.1088/0953-2048/25/2/025001

Critical current densities and n -values of MgB₂ strands over a wide range of temperatures and fields

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Table 1. Monofilamentary strand specifications and conditions.

Name	Process	MgB ₂ core ^a diam. (μm)	Dopant conc. (mol% C ^b)	Strand fill factor (%)	Heat treatment at soak
P0	CTFF	419 (d_0)	0	25.2	675 °C/20 min
P2	CTFF	325 (d_0)	2.09	15.2	675 °C/20 min
P3	CTFF	302 (d_0)	3.15	13.1	700 °C/20 min
I2	IMD	270 (d_0) 204 (d_i)	2.09	5.2	675 °C/30min

^a Mg:B atomic ratio, 1:2.

^b Based on C analysis by the LECO Corporation and normalized to the molar weight of MgB₂. No assumptions are made here concerning the expected uptake of C into the B sublattice; see, however, [15].

Table 2. Transport properties of the strands.

Strand	P0	P2	P3	I2
$J_{ct, 10\text{ K}}$ (10^5 A cm^{-2})	18.4	8.1	10.4	59.6
$B_{0, 10\text{ K}}$ (T)	1.07	1.86	2.30	1.90
$F_{p, \text{max}, 10\text{ K}}$ (GN m^{-3})	7.2	5.5	8.7	41.5
$F_{p, \text{max}, 4.2\text{ K}}$ (GN m^{-3})	5.6	8.4	11.3	60.9
Estimated connectivity, K (%)	12	9	15	69

$F_{p, \text{max}}$ reaches 60 GN/m³— it's like Nb₃Sn

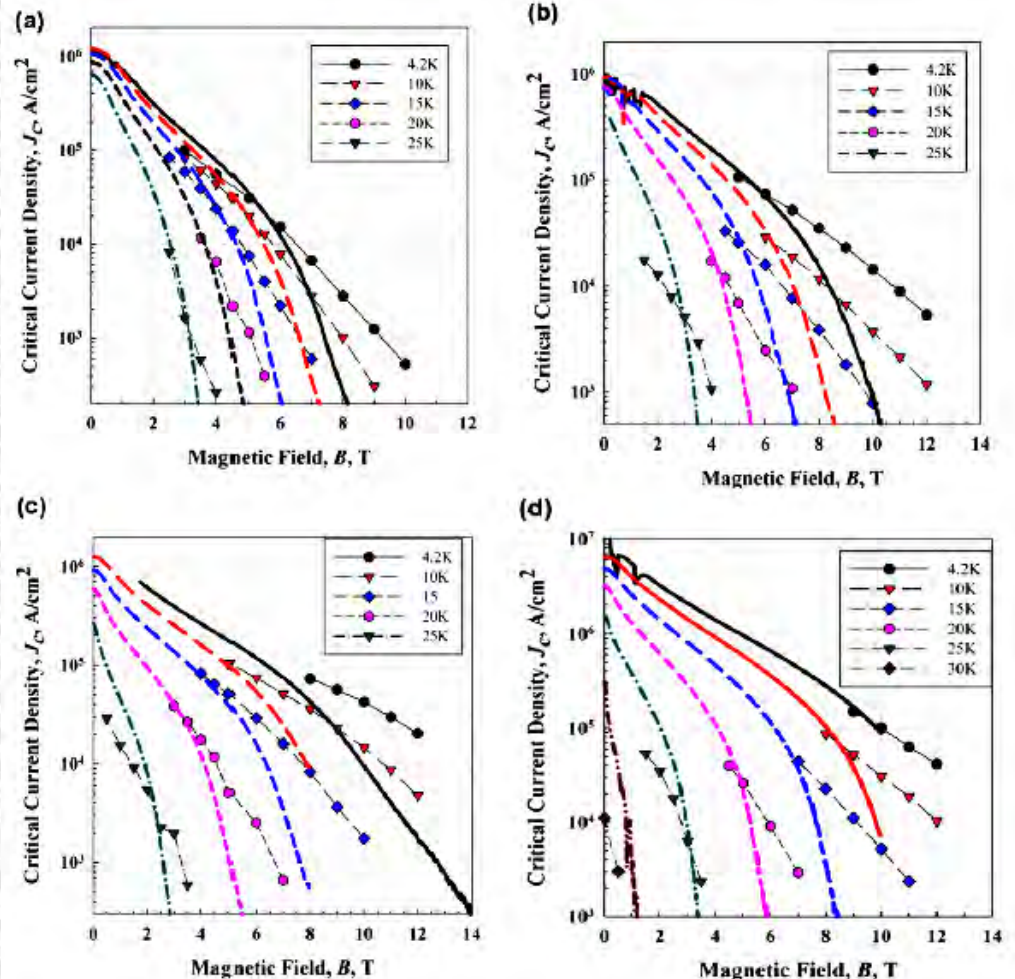


Figure 2. Transport J_{ct} and magnetic J_{cm} versus B in transverse applied fields at temperatures between 4.2 and 30 K for (a) strand P0 (undoped CTFF), (b) strand P2 (2% C-doped CTFF), (c) strand P3 (3% C-doped CTFF), and (d) strand I2 (2% C-doped IMD). The J_{ct} are represented by lines through data points; the J_{cm} are represented by the 'corresponding' full lines (arranged right-to-left in descending order of temperature).



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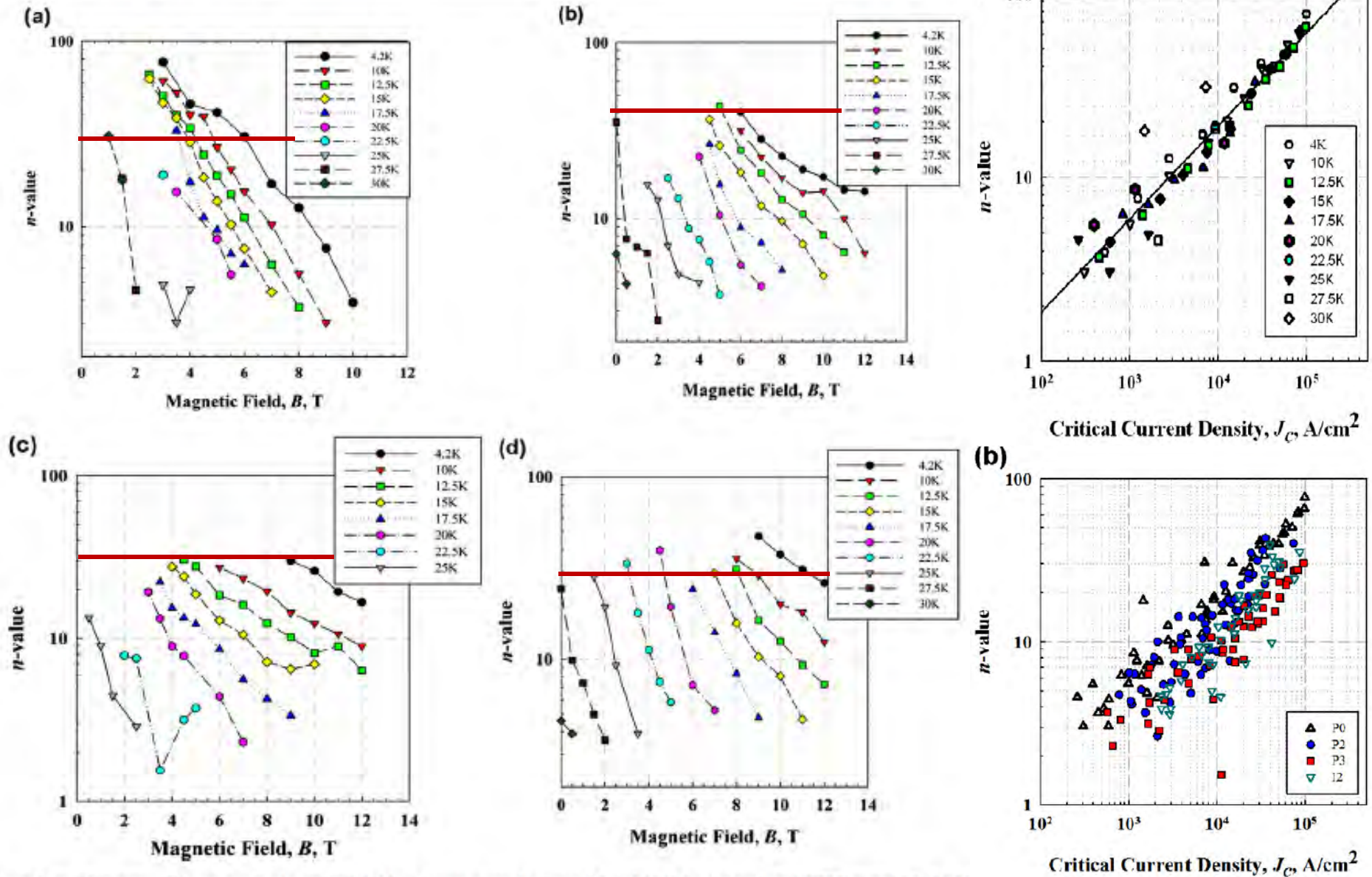
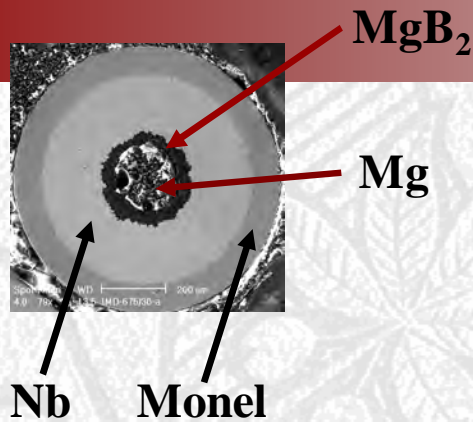


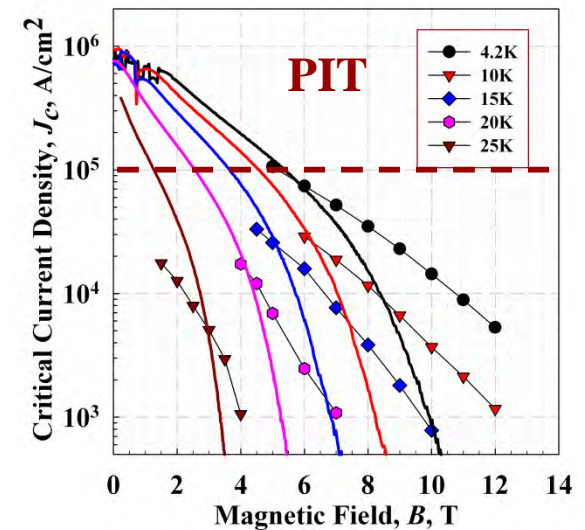
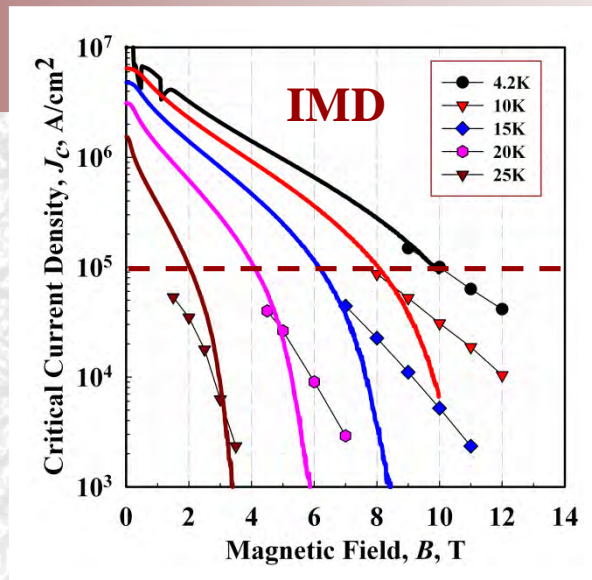
Figure 6. n -value versus B in perpendicular applied fields at temperatures between 4.2 and 40 K for: (a) undoped CTFF strand P0, (b) 2% carbon doped CTFF strand P2, (c) 3% carbon doped CTFF strand P3, and (d) 2% carbon doped IMD strand I2.

Motivation → Modification

1. Diffusion processed wire (Mg-RLI or IMD): initiated by Giunchi *et al.* (Edison SpA) – very dense, but low J_c because of powder type
2. Developed by Kumakura *et al.* (NIMS) – their B_c , much higher J_c s
3. Further developed at OSU-HTR – use of very fine B_c , and C-doping led to even higher J_c s
4. But – area of MgB_2 small – making this of more scientific than practical use. How to maximize conversion, and thus I_c ?



low MgB_2 fill factor so low J_c !



(G Z Li *et al.*, SUST, 2012)

■ high layer J_c . 1.0×10^5 A/cm² at 4.2 K, 10 T

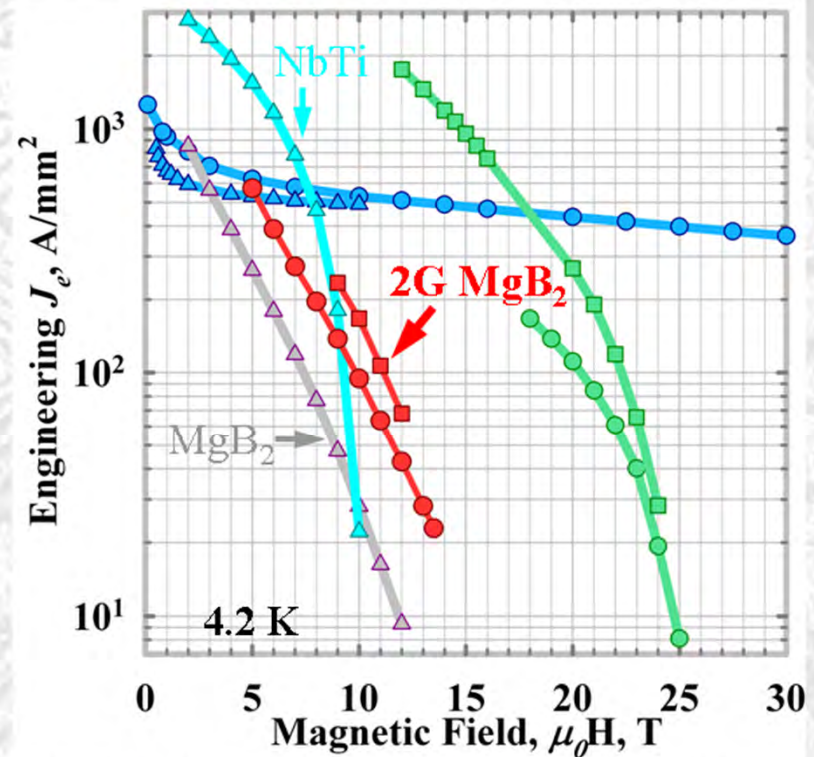
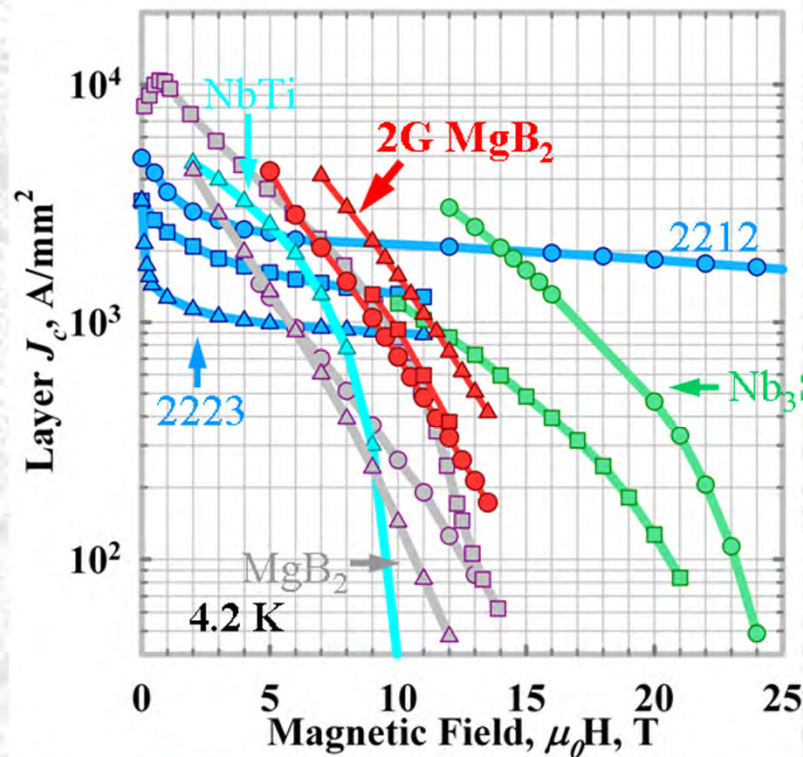


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A comparison to best-of Results

Second generation MgB₂ wires: both improved critical current density J_c and engineering J_e .



- 2G IMD Barrel
- 2G MgB₂ (2% C)
- ▲ 2G MgB₂ (3% C)
- △ PIT MgB₂ Wire
- MgB₂ Tape (Grasso)
- ▲ NbTi (Inoue)
- Nb₃Sn ITER
- Nb₃Sn (Internal Sn)
- ▲ 2223 (B-perpendicular)
- 2223 (B-parallel)
- 2212 Round Wire



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Samples

Table 1 Monocore wire Diameter and HT Conditions

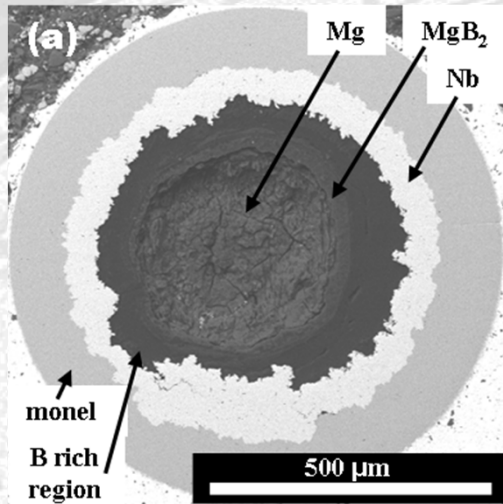
Sample No.	Diameter, mm	HT time at 675 °C, h
A1	0.83	0.5
A2	0.83	1
A3	0.83	8
B1	0.55	0.5
B2	0.55	1
B3	0.55	4

- 2% C doped plasma synthesized B (40 nm, Special Materials Inc.).
- Temperature: 675 °C, based on experience with PIT wires.
- Fabricated and heat treated at Hyper Tech Research, Inc.
- Transport J_c measurement at 4.2 K, up to 12 T.

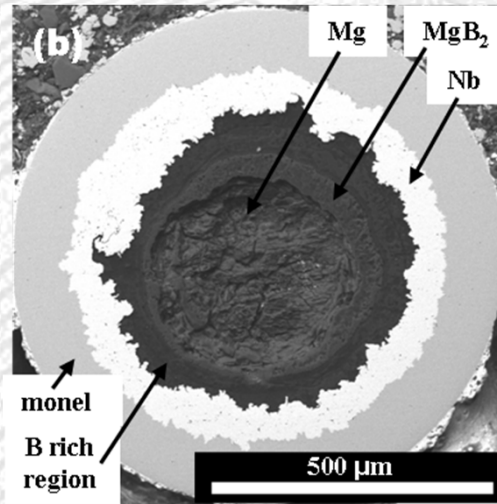
SEM & EDS: Quanta 200 with EDAX EDS system, FEI Sirion SEM.

A-series Infiltration-Processed Strands

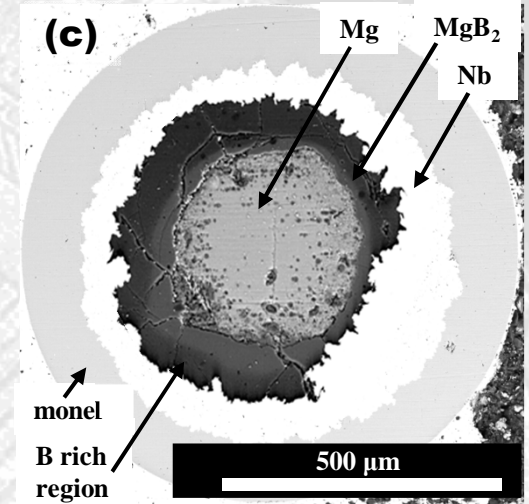
A1: 0.5 h



A2: 1 h

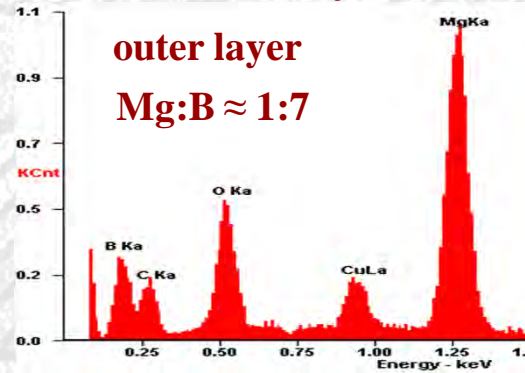


A3: 8 h

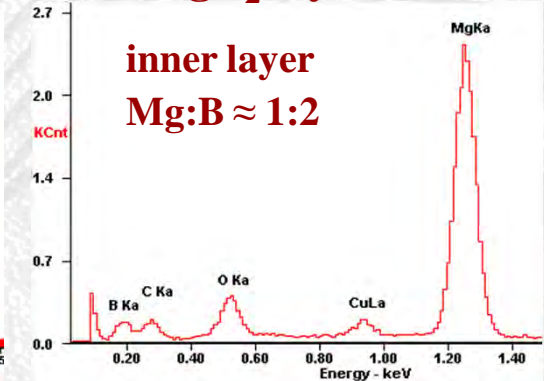


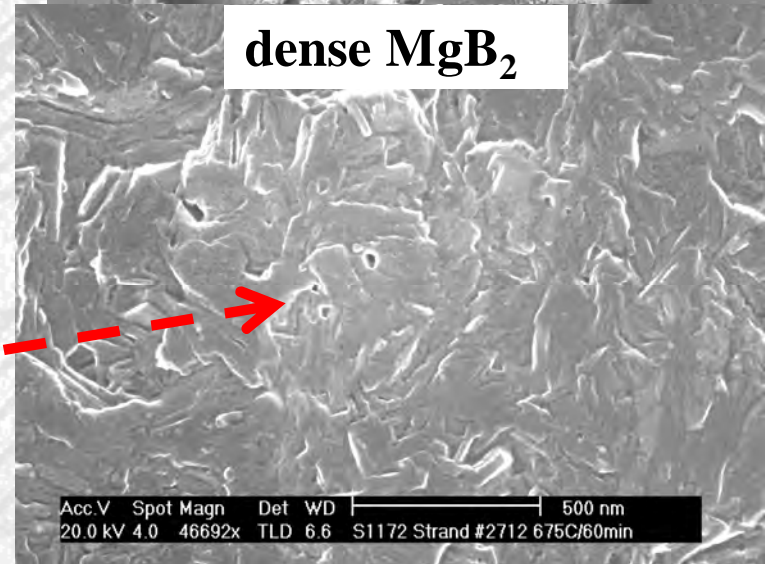
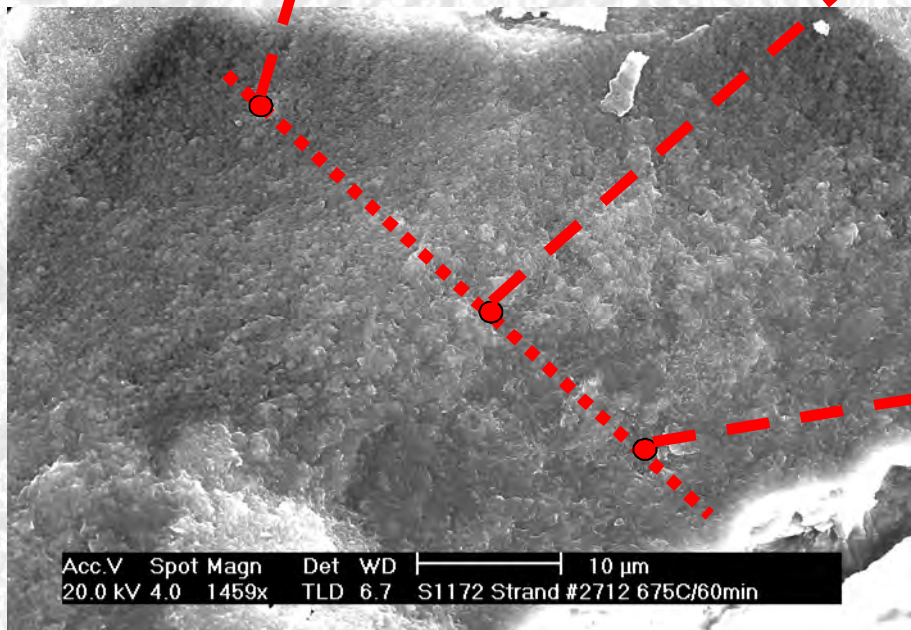
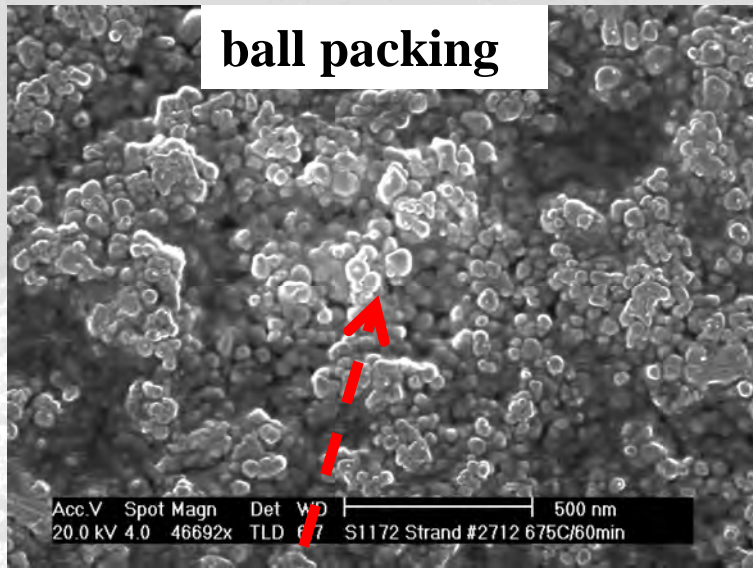
- MgB₂ circular layers are formed.
- MgB₂ composition and structure identified.
- B is not fully reacted even after 8 h at 675 °C.

B-rich layer

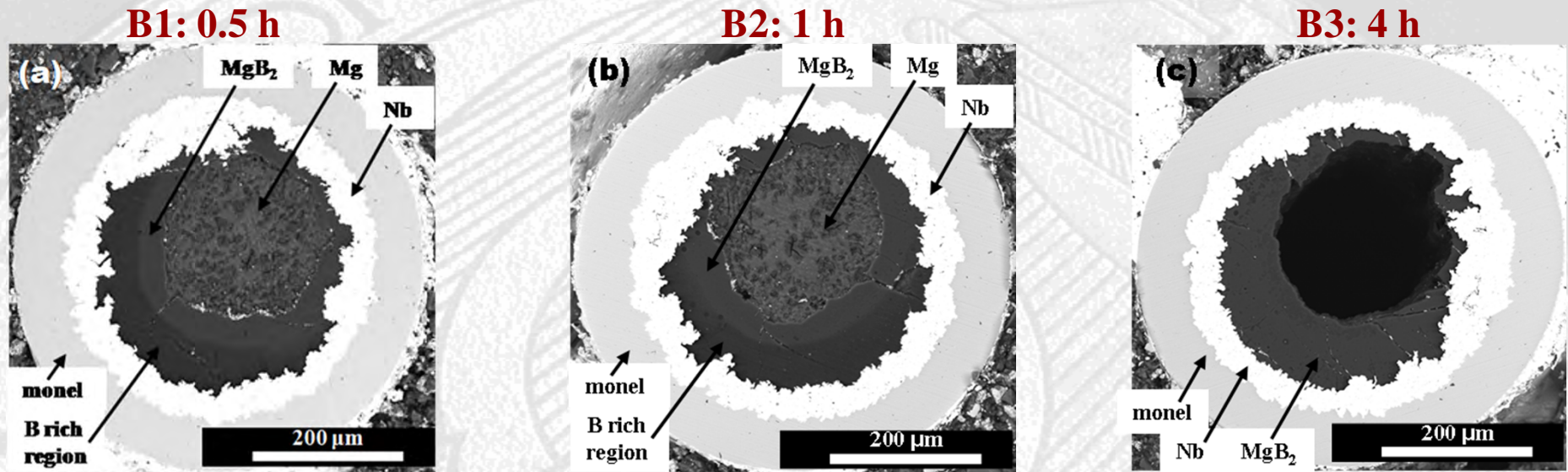


MgB₂ layer





B-series Infiltration-Processed Strands



- MgB_2 circular layers are formed but the Mg rod is off-centered.
- B is fully reacted after 4 h heat treatment at 675 °C.
- MgB_2 area and layer thickness are listed in table 2.

Comparison of A- & B-series Strands

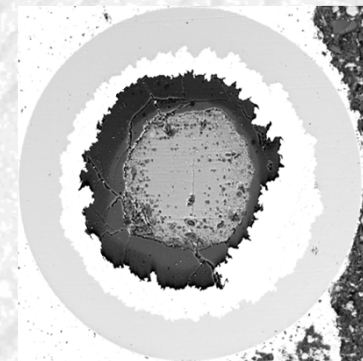
Table 2: MgB₂ Layer Thickness, Areas and Corresponding Fill Factors

Sample No.	HT time at 675 ° C, h	MgB ₂ layer thickness, μm	MgB ₂ area, μm ²	MgB ₂ fill factor, %*	MgB ₂ + B fill Factor, %**
A1	0.5	0 ~ 30	26900	4.4	21.1
A2	1	10 ~ 50	38200	6.3	19.7
A3	8	15 ~ 60	44800	7.5	19.1
B1	0.5	0 ~ 40	25100	10.1	19.6
B2	1	0 ~ 49	28000	11.2	19.7
B3	4	0 ~ 90	46700	18.8	18.8

* Area fraction of MgB₂ in the overall transverse cross section of the wire

** Area fraction of MgB₂ plus unreacted B powder after heat treatment

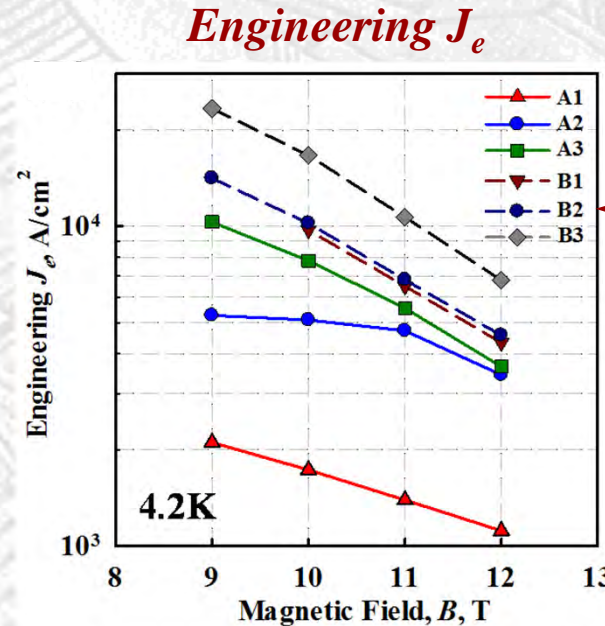
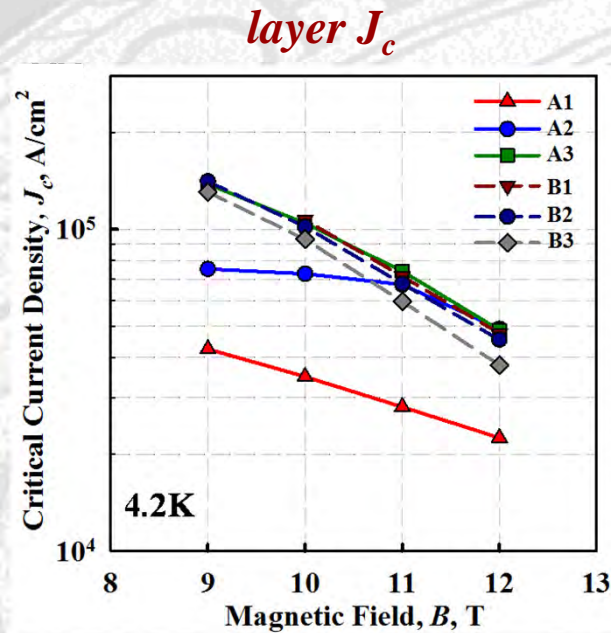
- MgB₂ layer thickness increases as HT time increases.
- MgB₂ fill factor increases as HT time increases.



Layer J_c & Engineering J_e

$layer J_c = I_c / \text{MgB}_2 \text{ area}$

$J_e = I_c / \text{whole area}$



**B3: 1.67×10^4
A/cm² at 10 T**

All wires quench when $B < 9$ T.

▪ **layer J_c :**

A-series HT time \uparrow , $J_c \uparrow$; A3: 1.04×10^5 A/cm² at 10 T

B-series HT time \uparrow , $J_c \downarrow$; B1: 1.07×10^5 A/cm² at 10 T

▪ **Engineering J_e :**

B-series is better than **A-series** because of its high MgB₂ fill factor.



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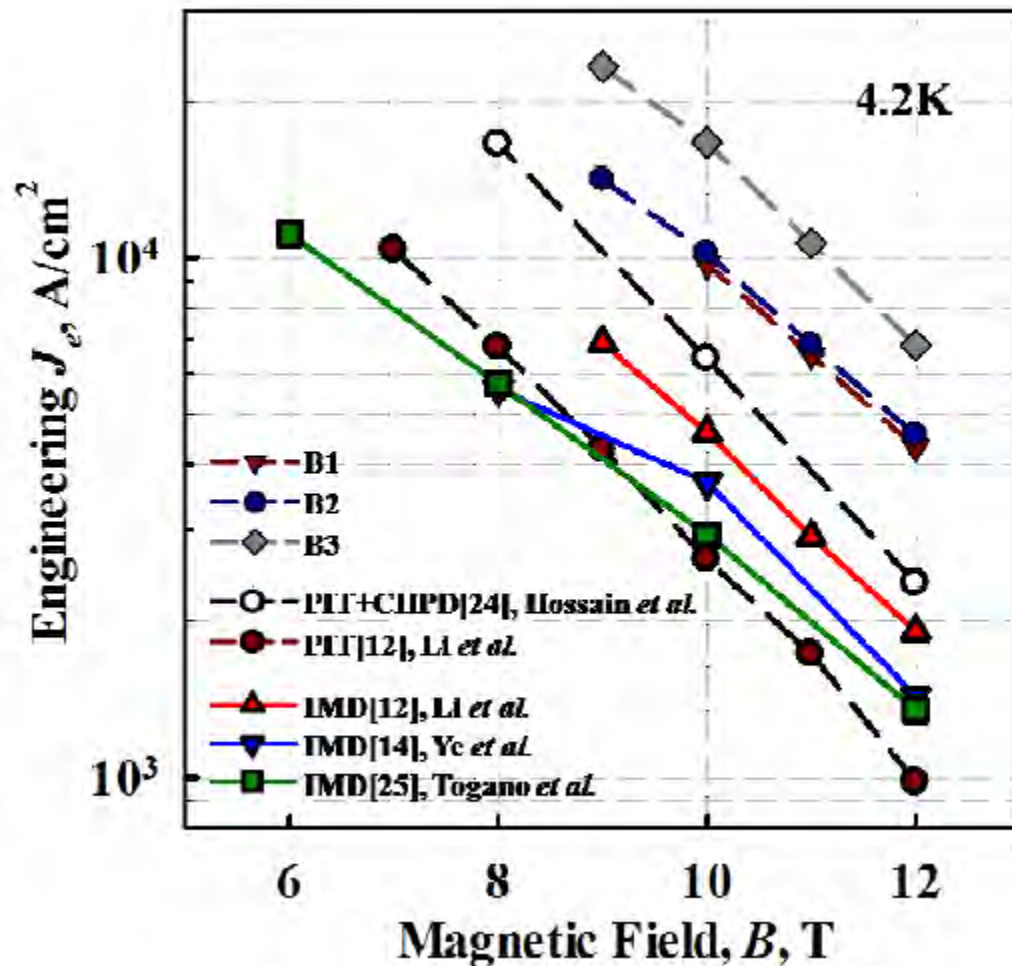
So, layer J_c is 10 x for 2G (and IMD), but How do the J_e results compare to PIT?

We have to correct for doping level and special processing

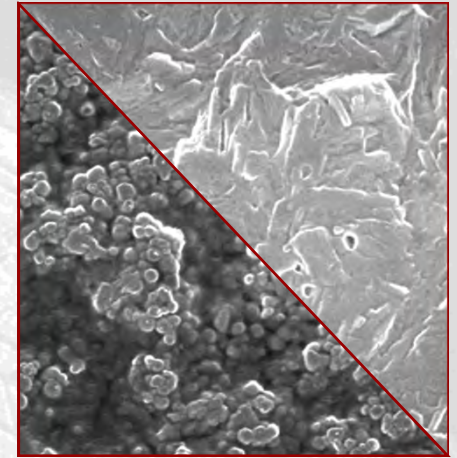
A head to head comparison is shown by the red filled circles and the gray diamonds - about a factor of 5 in J_e !

Pressure processing (sequential pressing, Flukiger/Hossain) on the same PIT wires is also shown (open circles)

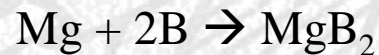
We have found 3-4% C can give high J_c s in PIT wires, now implementing in IMD



MgB₂ Layer Growth Mechanism



- **Step I: Melting of the Mg rod**
- **Step II: Liquid Mg infiltration and reaction with B**



B particles volume expansion are ~ **90%** considering: (a)
and (b) **V_{MgB_2} : 17.46 cm³/mol**

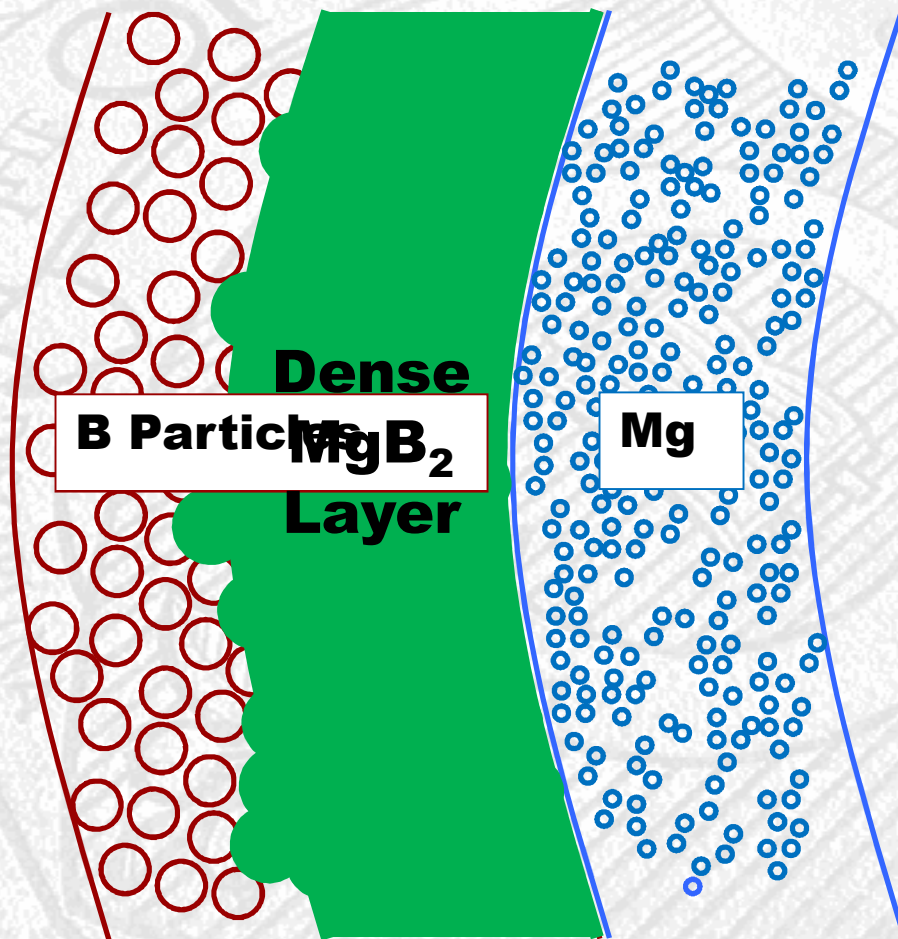
V_{B} : 4.59 cm³/mol

A dense MgB₂ circular layer is formed.

- **Step III: Mg atomic diffusion through the dense MgB₂ layer**

Atomic GB diffusion: Quite slower process than liquid Mg infiltration in step II.
Grain growth during long HT time, resulting in reduced layer J_c .

Demonstration: MgB_2 Layer Growth Mechanism



Melting of Mg

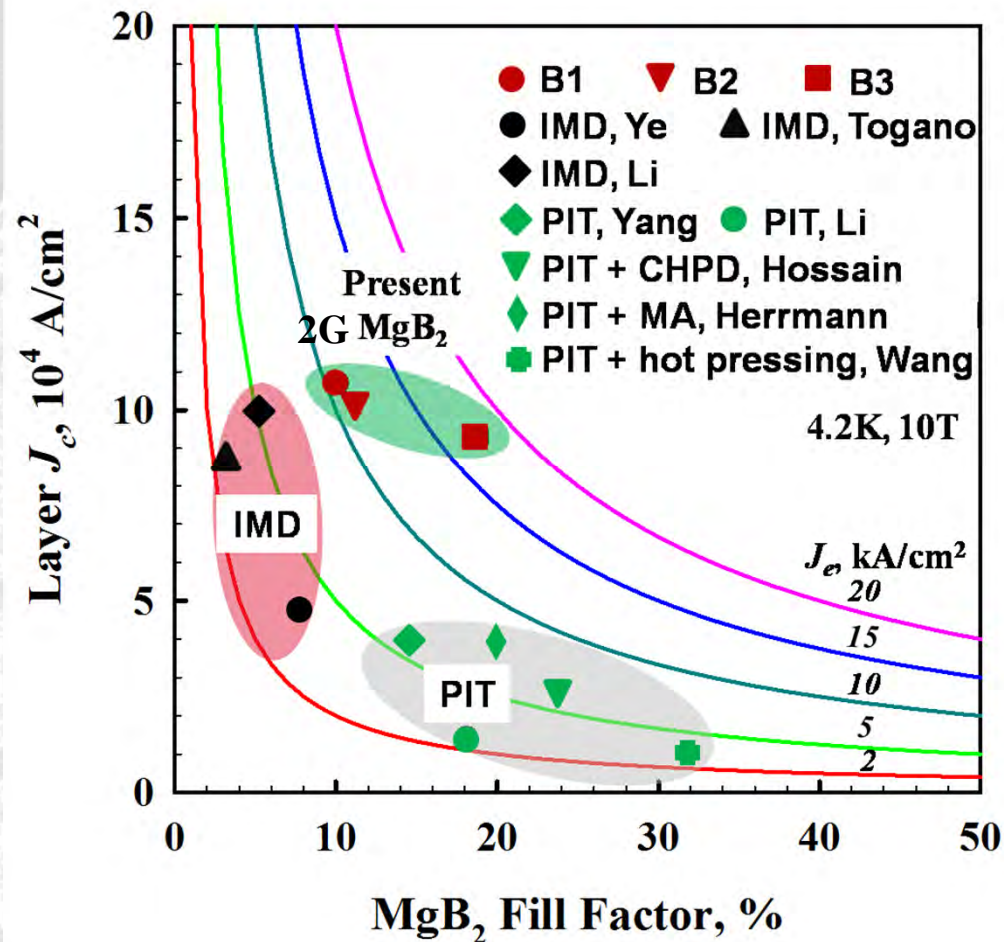


Liquid Mg Infiltration
& Reaction



Dense MgB_2 Formation
& Mg Atoms Diffusion

Principle to Design a Well-Performed Wire



Directions to obtain a good conductor:

J_c improvement.

- doping
- pressing
- ball milling
- infiltration/diffusion method

MgB₂ fill factor.

- smaller diameter wire
- multifilamentary wire

$$\text{layer } J_c = \frac{J_e}{\text{MgB}_2 \text{ fill factor}}$$

(S J Ye *et al*, Physica C, 2011; K Togano *et al*, SUST, 2009; G Z Li *et al*, SUST, 2012; Y Yang *et al*, IEEE Trans Appl Supercond, 2012; M Hossain *et al*, SUST, 2009; M Herrmann *et al*, APL, 2007; D L Wang *et al*, SUST, 2012)



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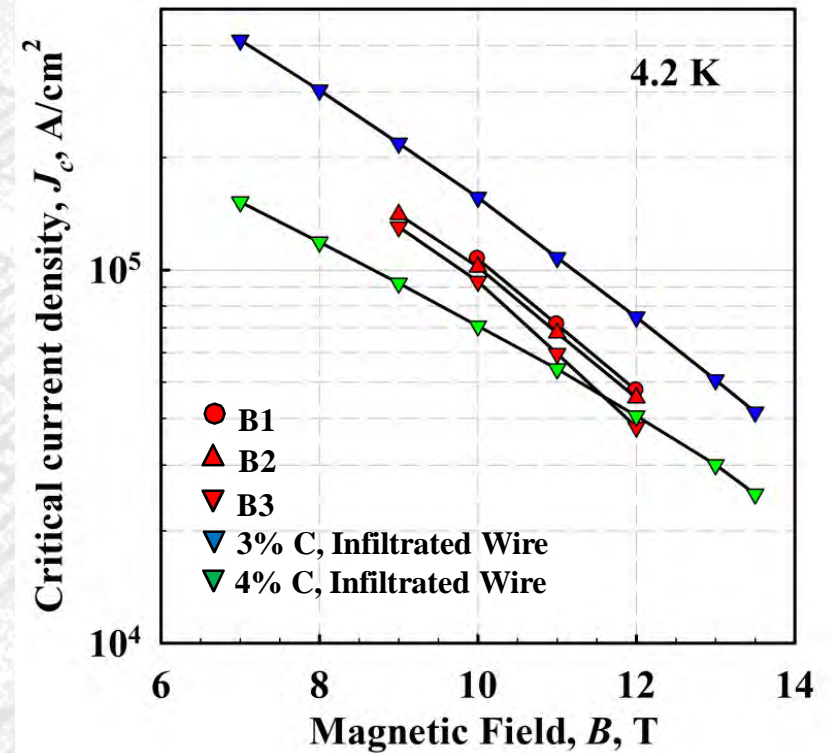
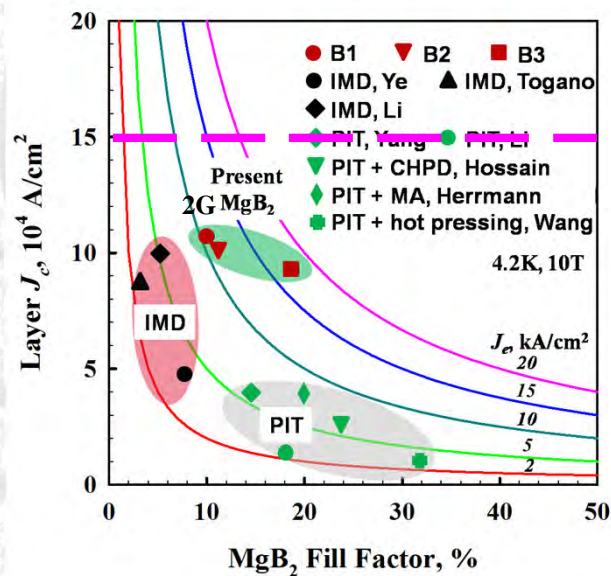


Further Explorations I: layer J_c enhancement

Carbon doping level

3% and 4% C doped diffusion processed strands, heat treated at 675 C, 4H.

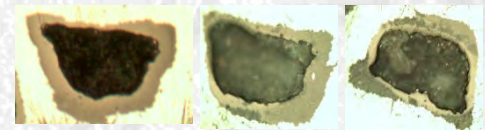
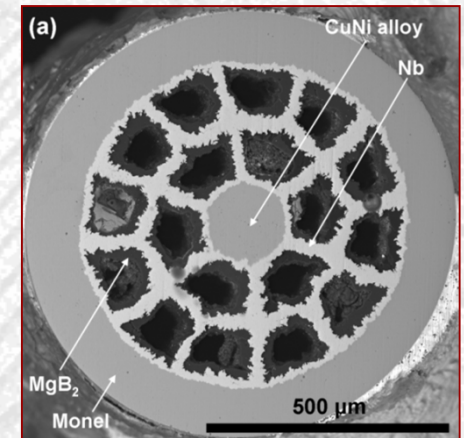
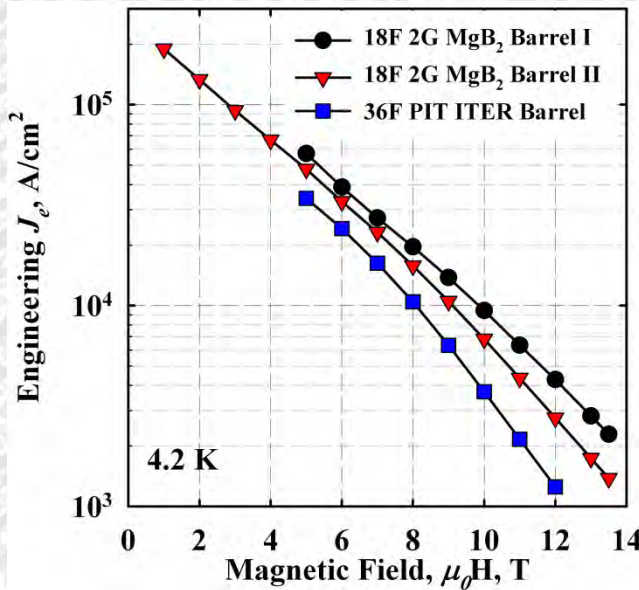
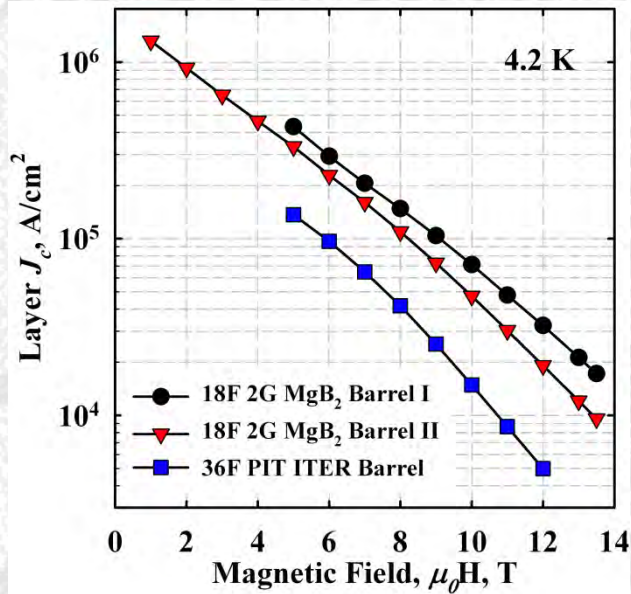
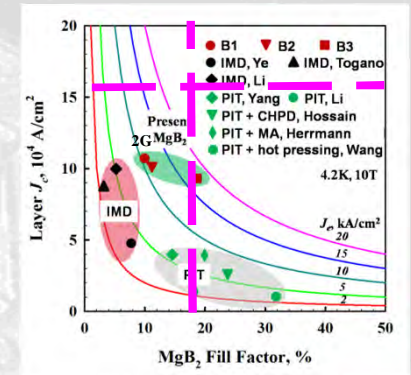
Best layer J_c for 3% C doped diffusion processed strands: 1.57×10^5 A/cm² at 4.2 K, 10 T.



Further Explorations II: Multifilamentary 2G Wires

■ 2G MgB₂ multifilamentary long strands (ITER barrel).

Layer J_c and J_e higher than typical multifilamentary PIT barrels, but lower than our best 2G monocore short wires.



N-values in PIT MgB_2

- Improvements in 4 K values
- Improvements in n values as a function of B and T

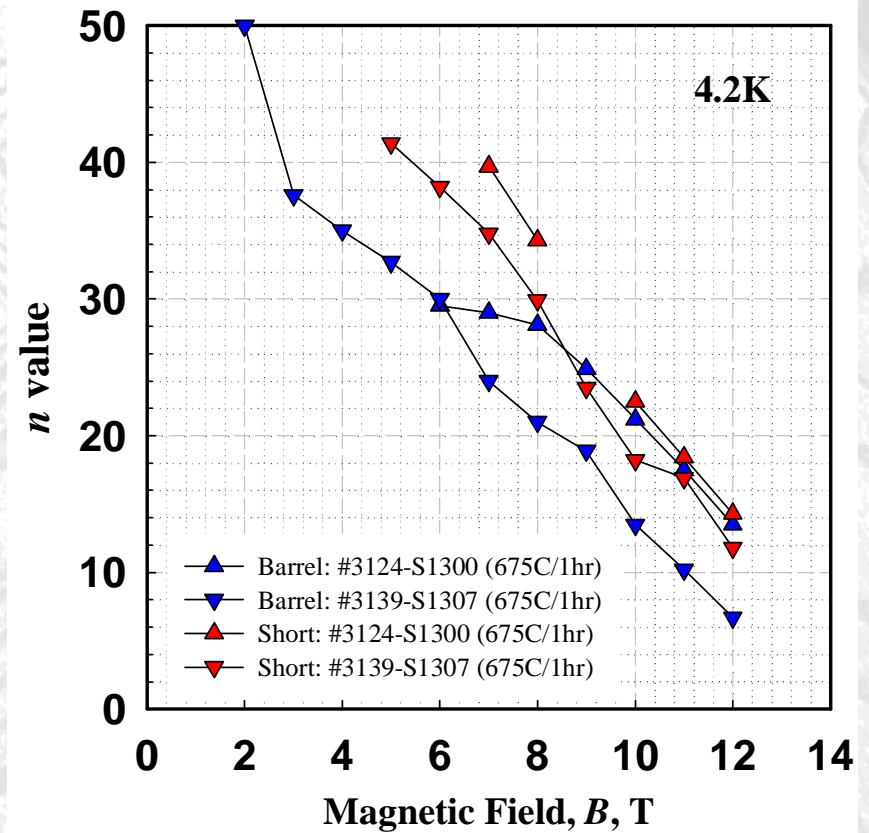
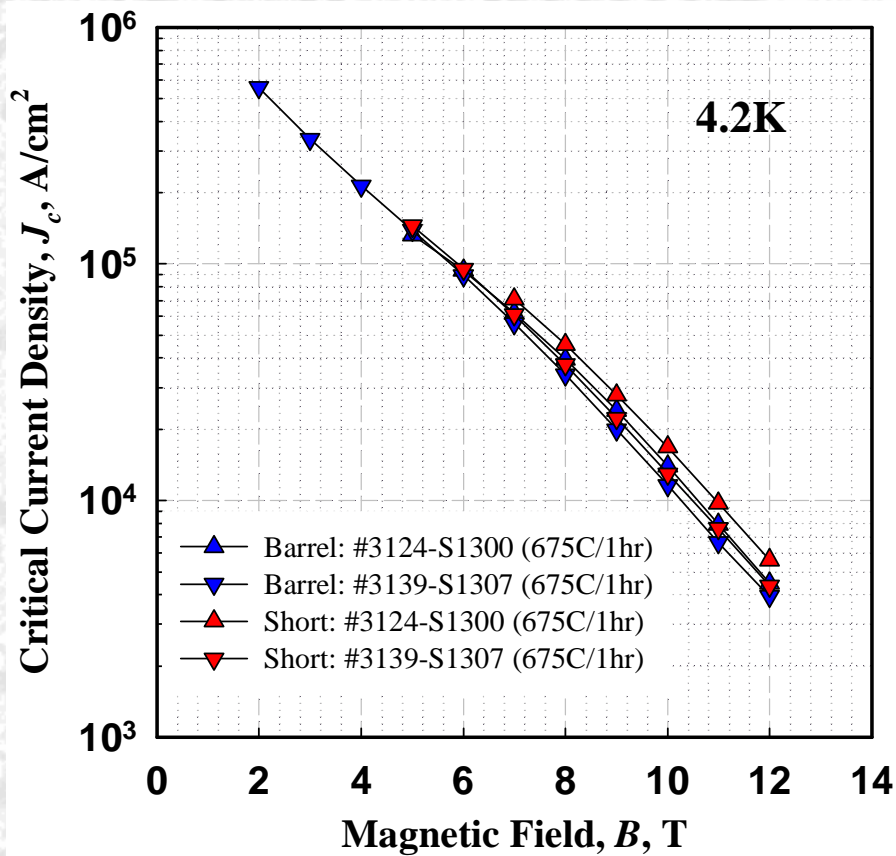
Strand Types

Strand #	# Mono	Barrier	Mono sheath	Multi sheath	Central fil(s)	powder material	dia (mm)	% powder	HT (°C/min)
3139	24	Nb	-	Monel	Cu	MgB2_2%C†	0.83	11.7	675 / 60
3139	24	Nb	-	Monel	Cu	MgB2_2%C†	0.83	11.7	700 / 60
3124	30	Nb	-	Monel	Cu	MgB2_2%C	0.83	23.3	675 / 60
3124	30	Nb	-	Monel	Cu	MgB2_2%C	0.83	23.3	700 / 60

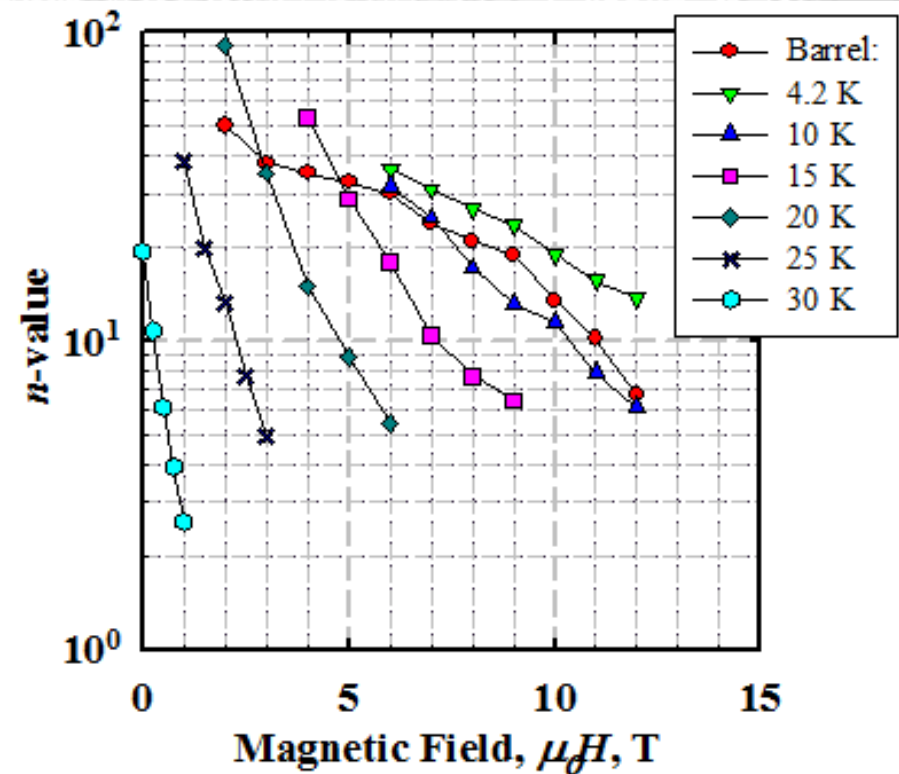
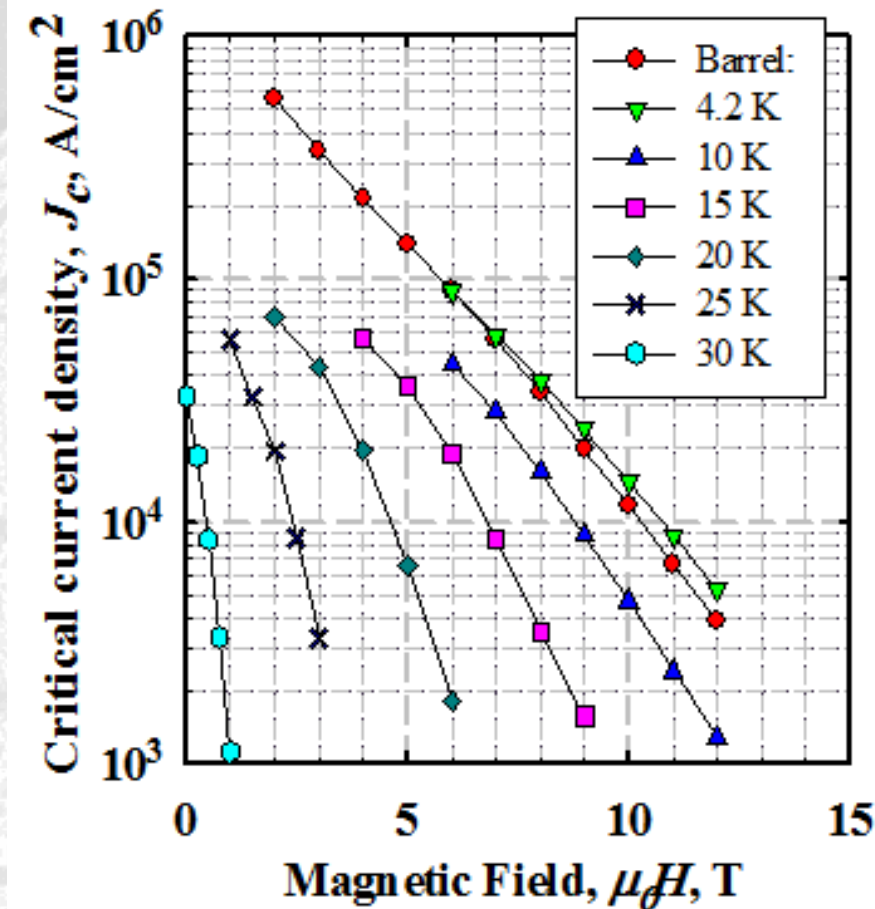
Early Barrel Study

3139-S1307	24	Nb	-	Monel	Cu	MgB2_2%C†	0.83	11.7	675 / 60
3124-S1300	30	Nb	-	Monel	Cu	MgB2_2%C	0.92	23.3	675 / 60

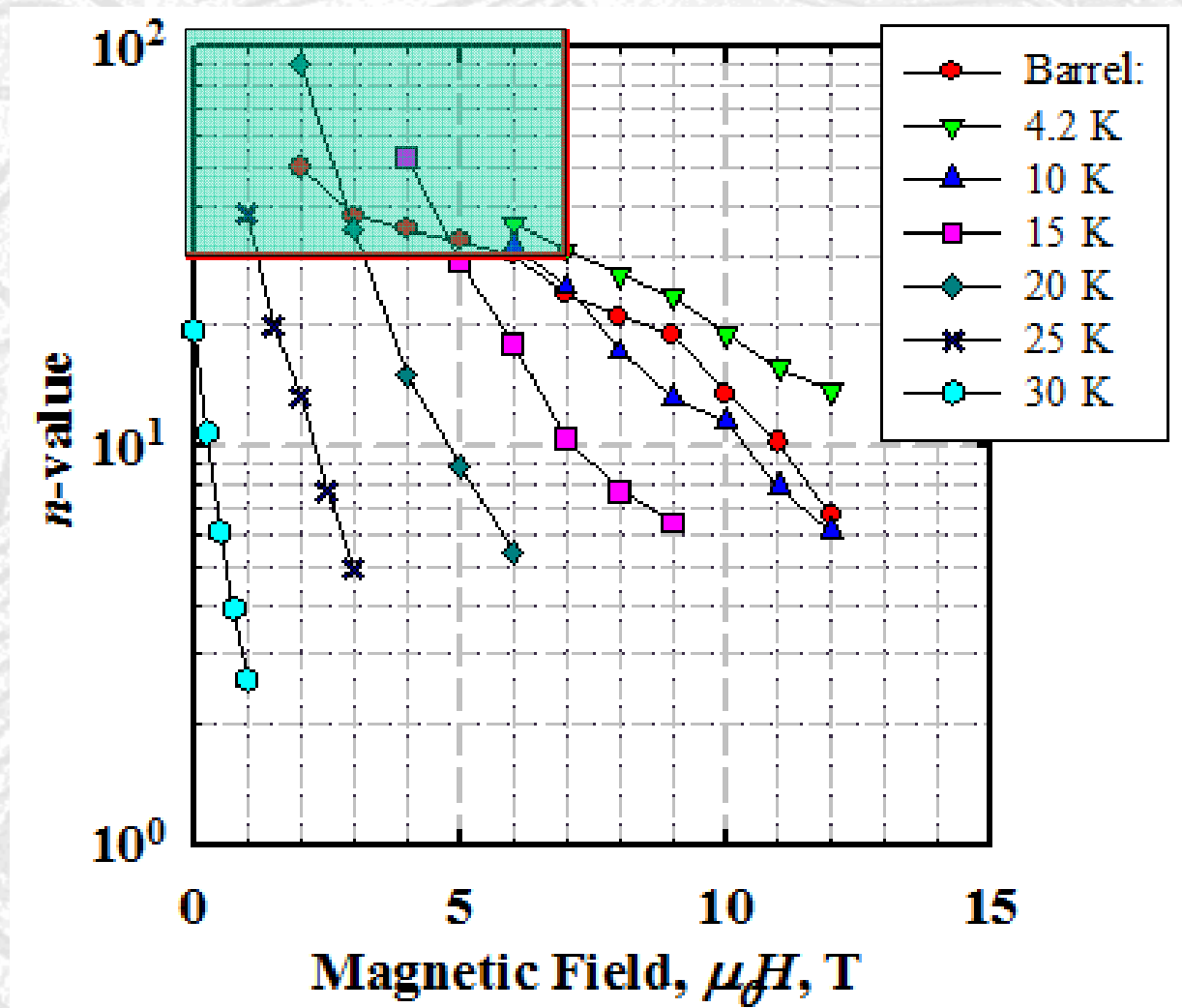
J_c and n value at 4.2K



24 Filament (3139-S1307)



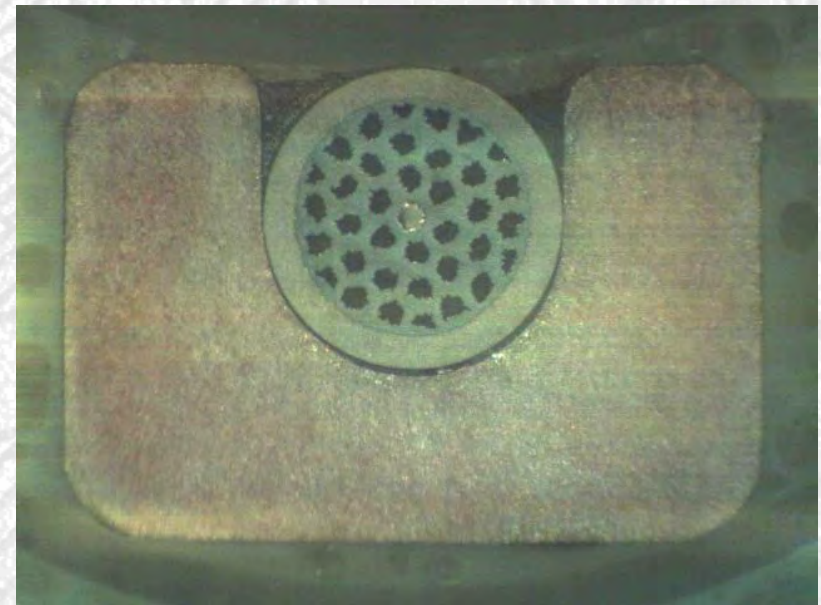
Regimes of n-value



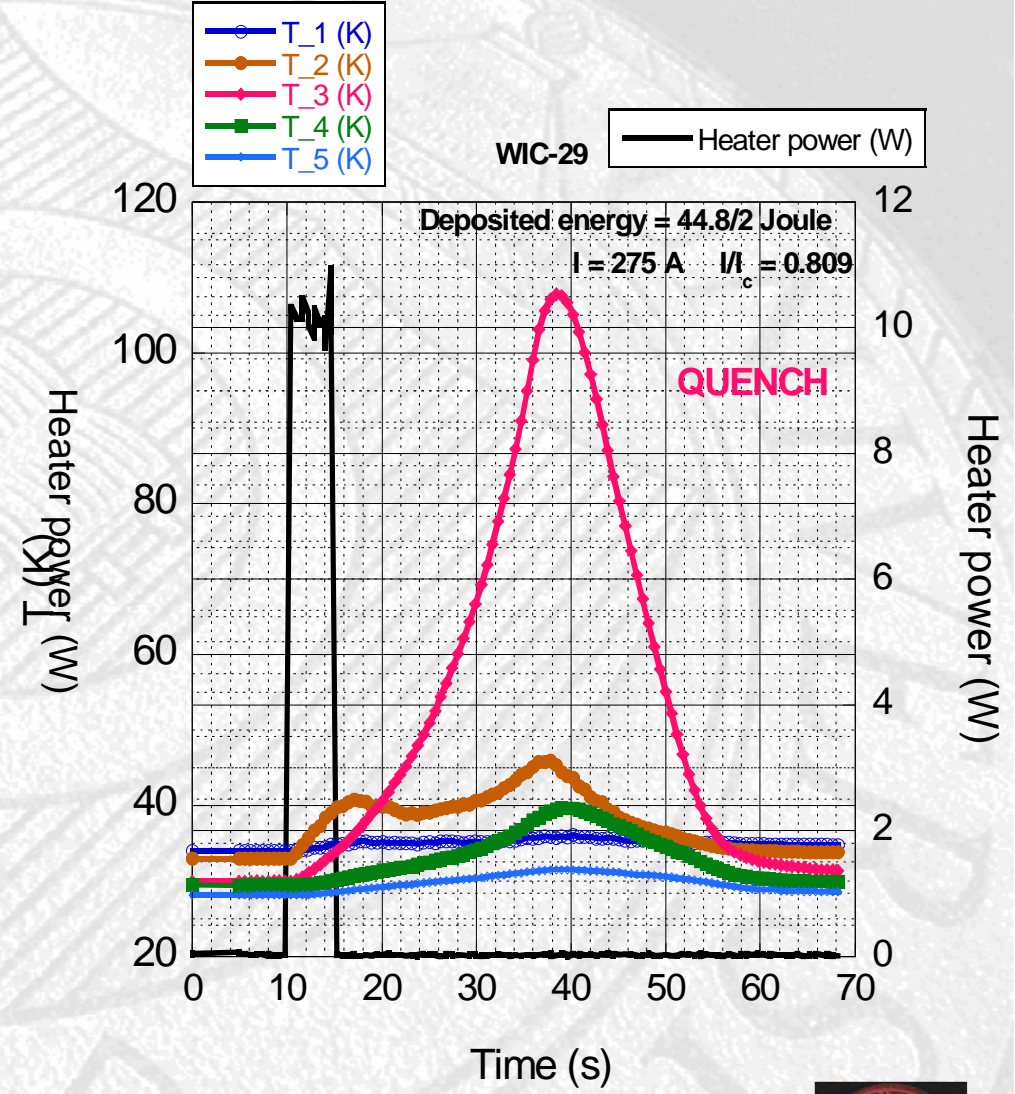
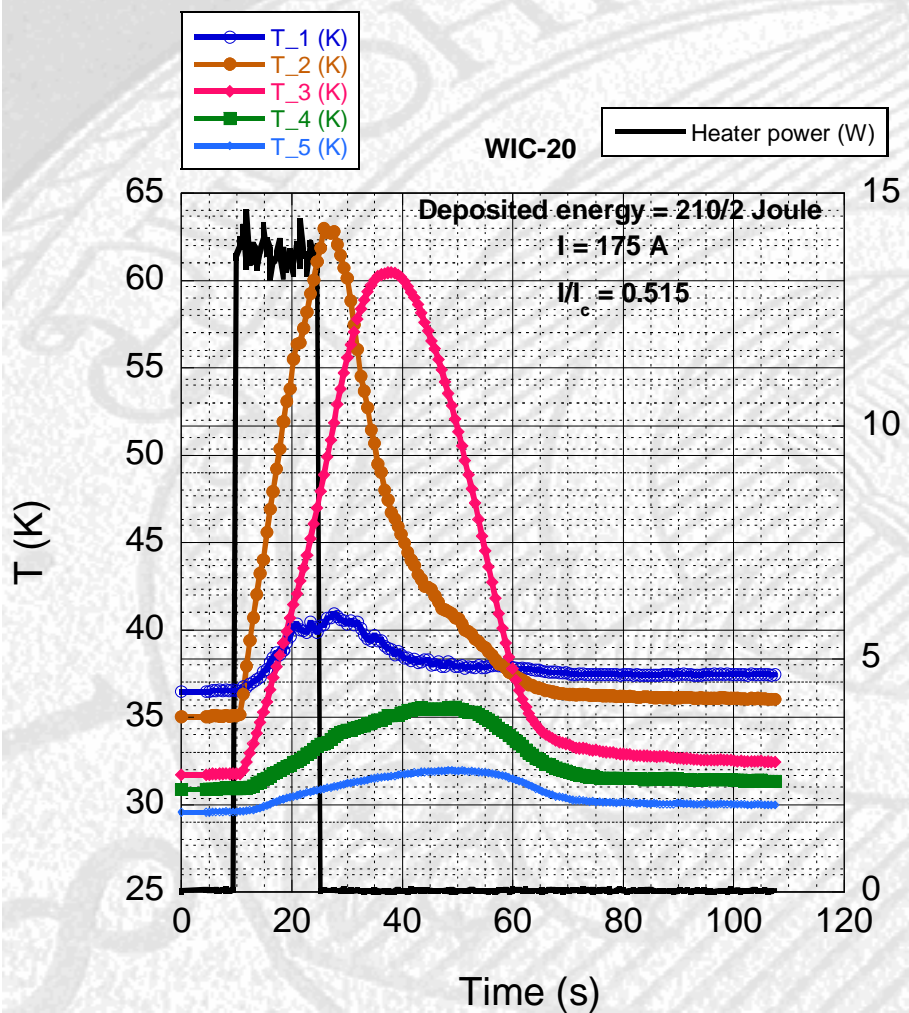
N > 30
4K 7 T
10K 6 T
15K 5 T
20K 3 T
25K 1 T

WIC and Coil Test

- WIC Test
- Coil Manufacture and Test



WIC NZP and Quench (gas cool)



NZP: 0.5-1 cm/s



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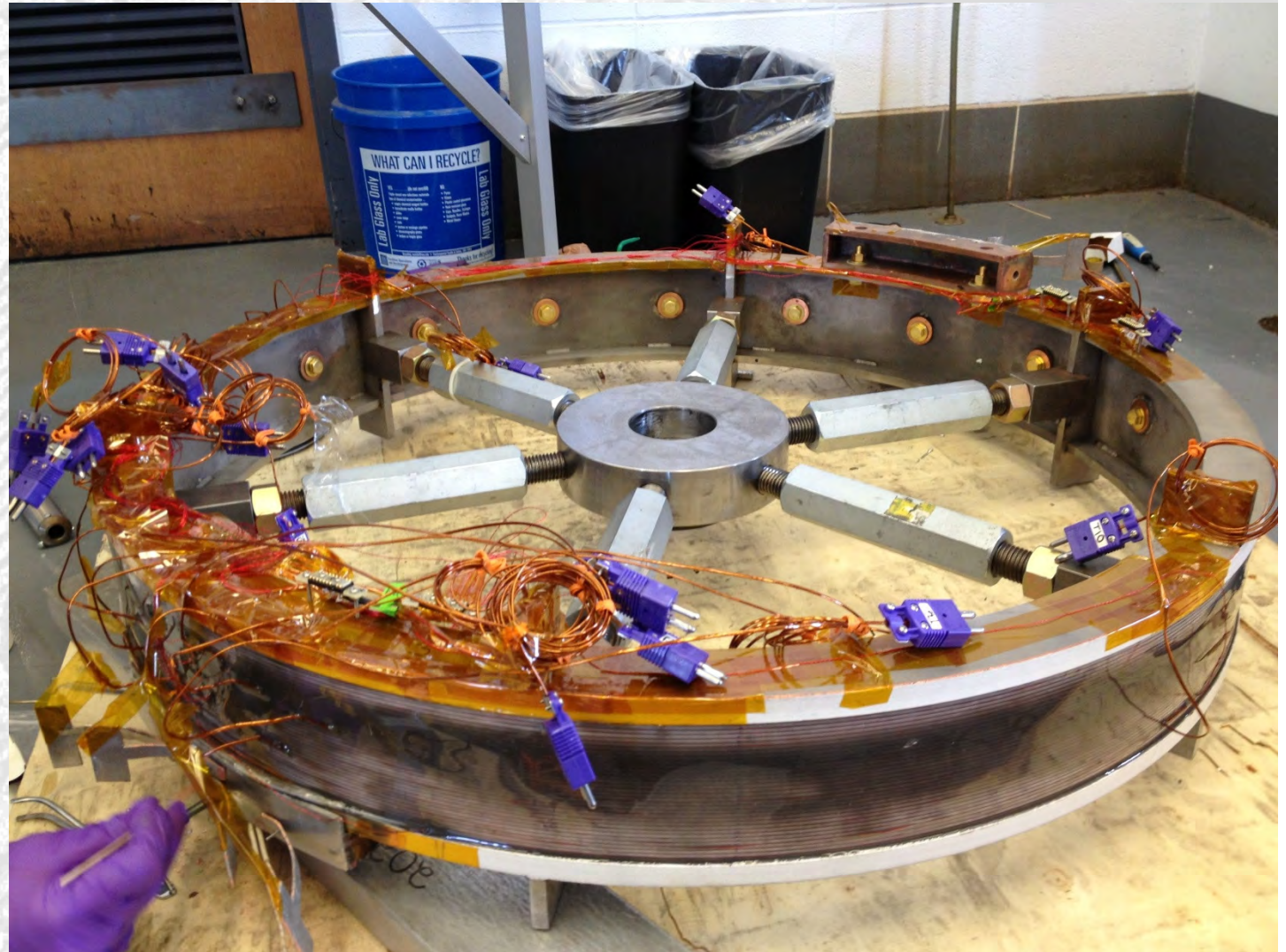
MgB₂ Coil, 100 m of WIC MgB₂ Conductor

HTR: MgB₂ strand, Wire-in-channel Conductor

HTR: Coil wound, coil epoxy impregnated by HTR

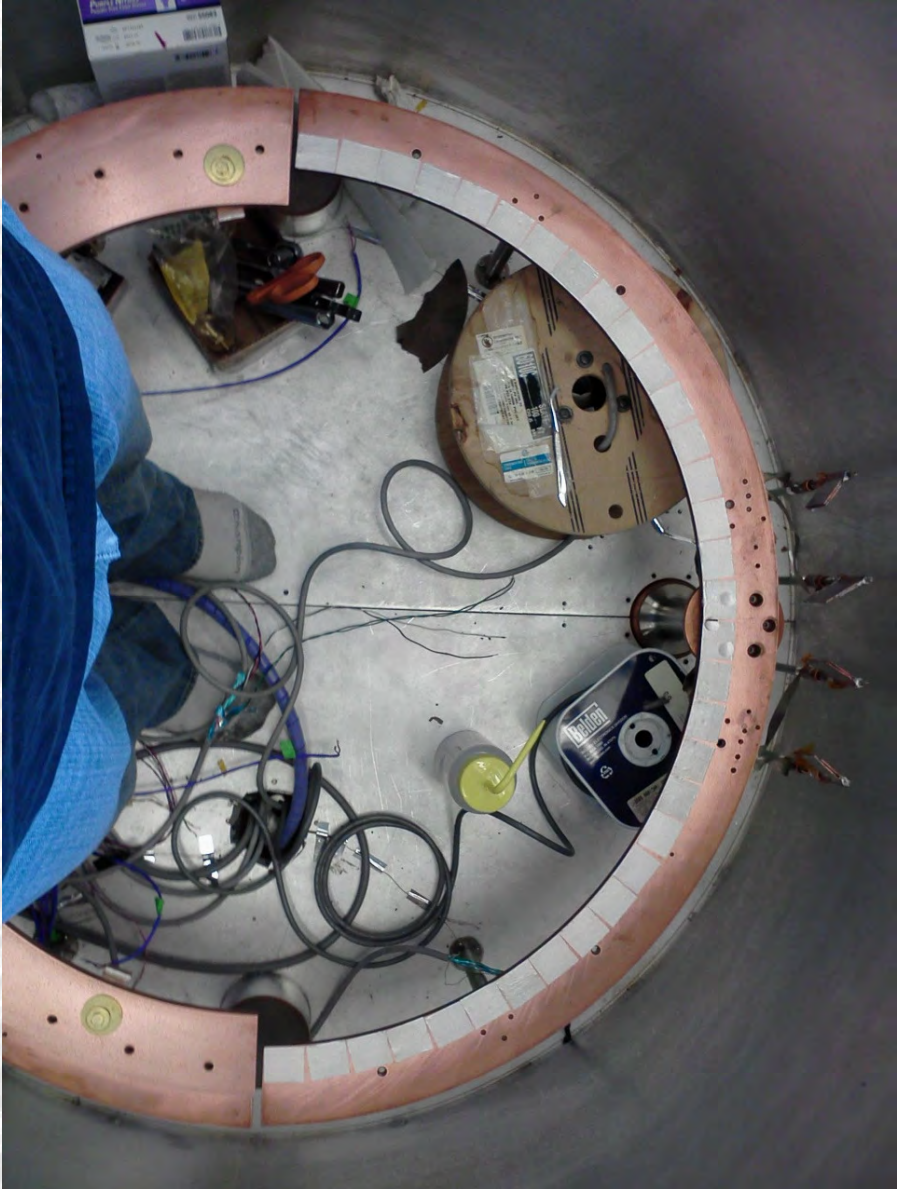
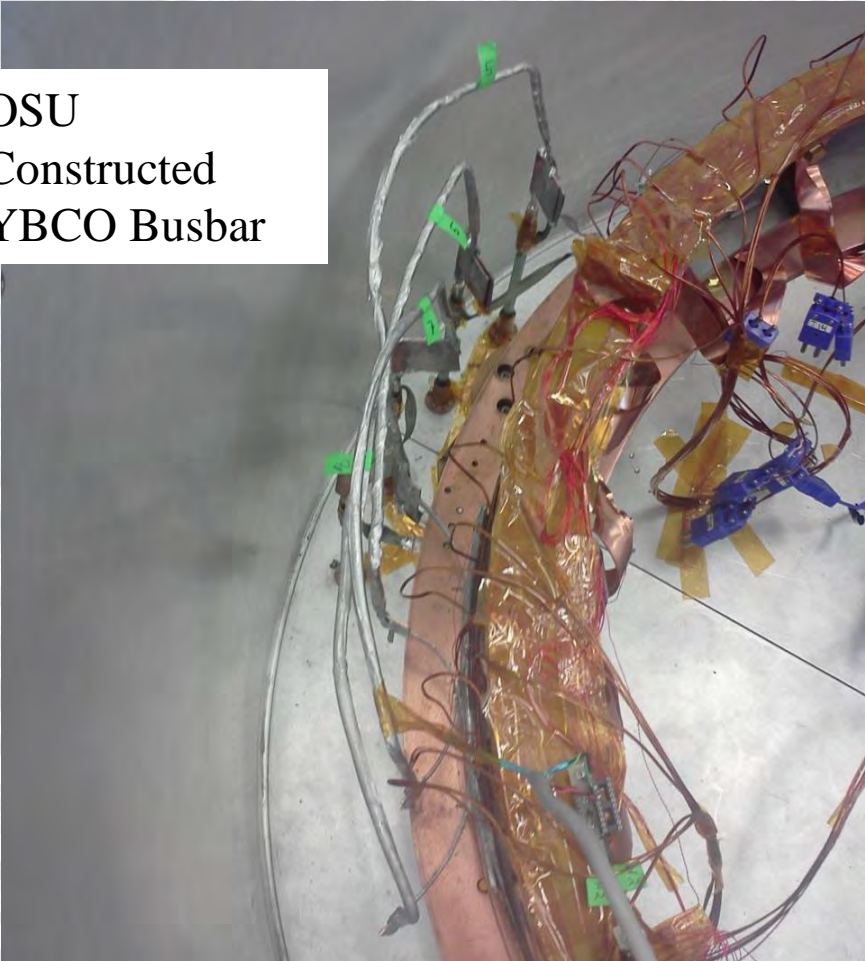
OSU: Coil Instrumented with 30+ voltage taps, 18+ thermocouples, other sensors

OSU: Cool down and Test

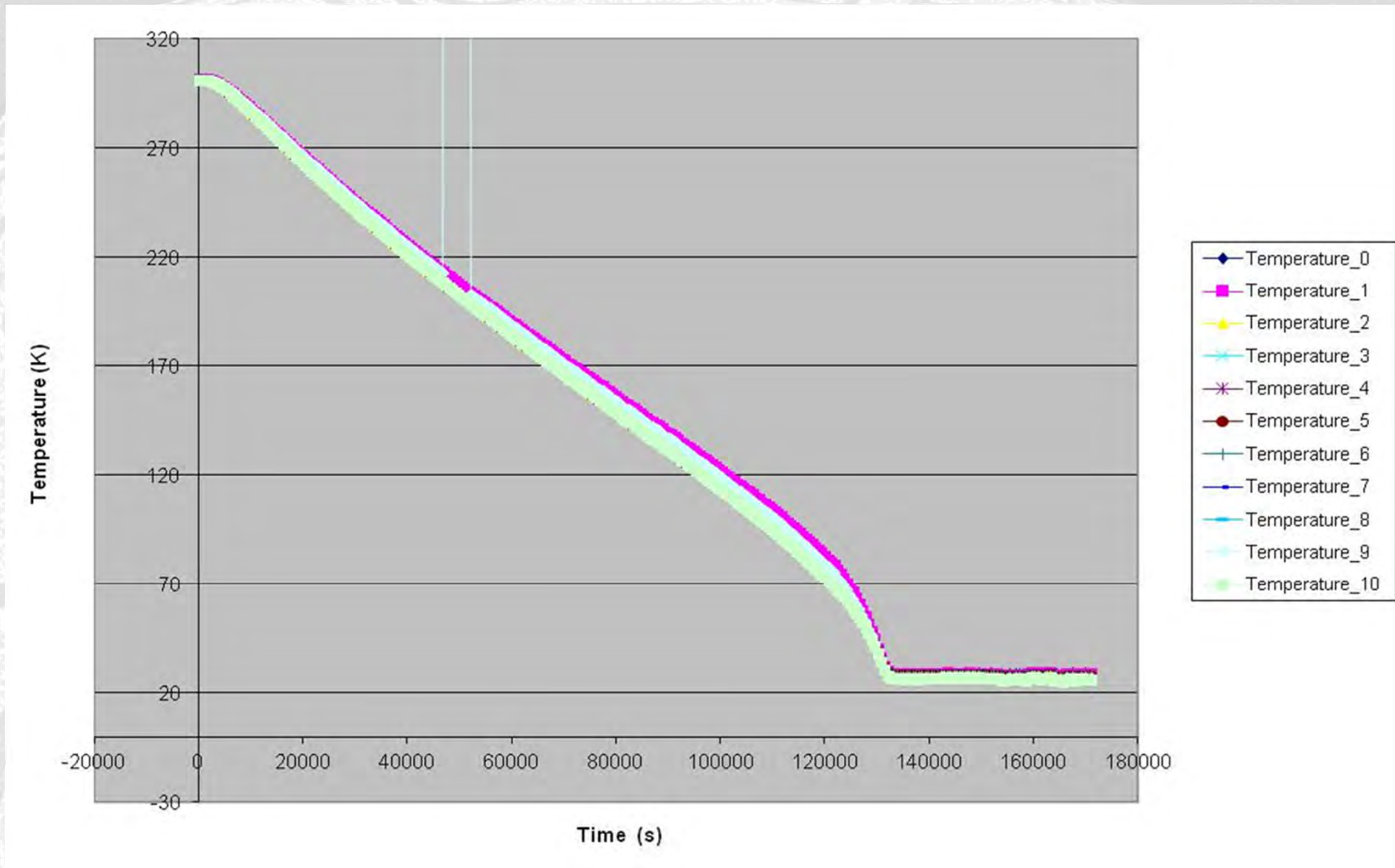


Inside of OSU Dewar

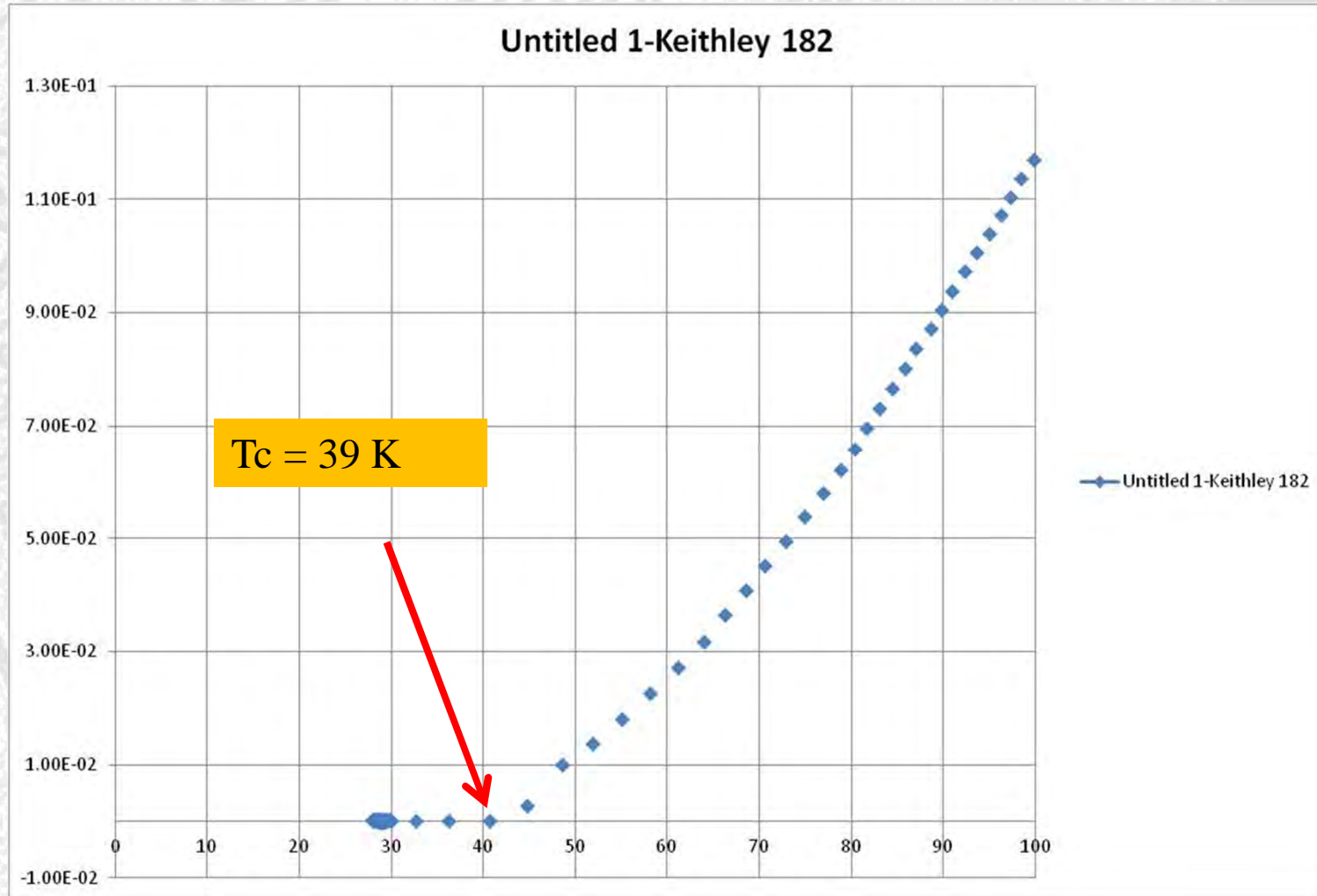
OSU
Constructed
YBCO Busbar



Initial Coil Cool-down

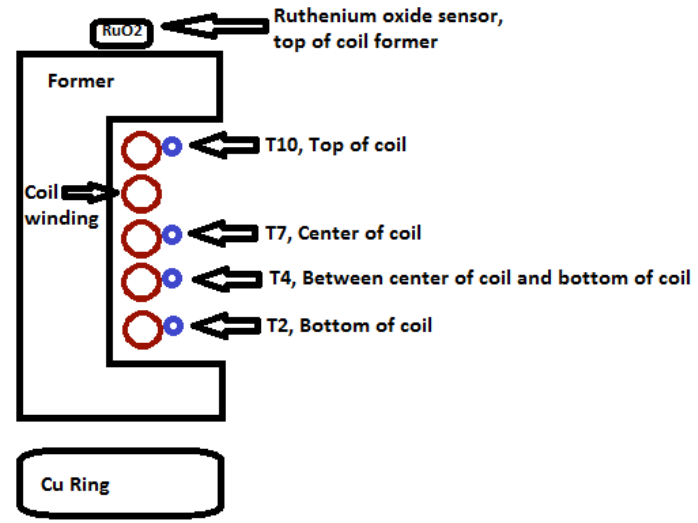


T_c transition for coil



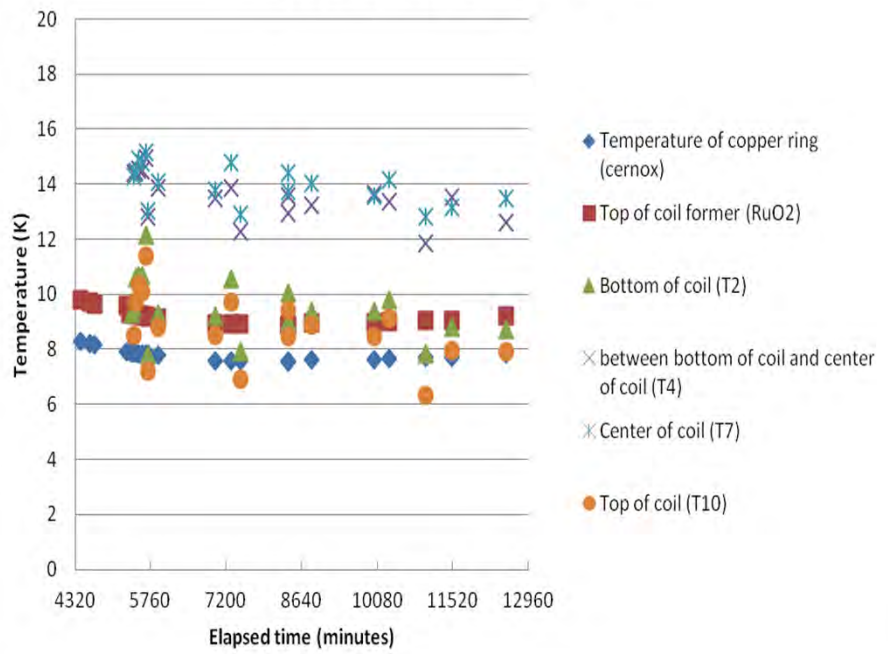
Coil T Instrumentation / Result

Axis of symmetry

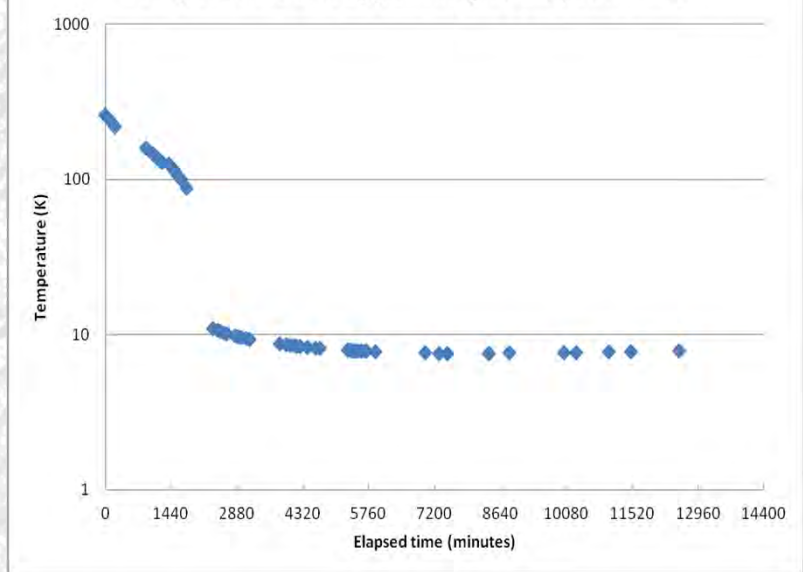


- Thermocouple
- Coil winding

Temperature vs Elapsed Time of Various Locations Around Coil



Temperature of Copper Ring vs Elapsed Time



Summary

- Percolation-based models were used to compare PIT and IMD MgB₂ wires, and it was found that previous treatments which attributed all high field J_c loss to anisotropy were overly pessimistic, with about 40% (or a large reduction) due to porosity
- This led to an impetus to push IMD from “scientifically interesting” (high J_c (10 X PIT), low J_e) to practical conductors
- These considerations led to advanced, “2G”, MgB₂ wires were fabricated by incorporating (1) a Mg diffusion method, (2) fine B powder, (3) C-doping at the powder level, (4) Mg/B ratio, and (5) strand geometry modifications.
- 2G MgB₂ wires had dense MgB₂ layer structures, enabling best layer J_c of 1.0×10^5 A/cm² at 4.2 K and 10 T (***an increase by a factor of 10!***)
- The best J_e of sample B3 achieves 15.7 kA/cm², in increase of about 5 from the same chemistry PIT conductors, and ***a factor of 3 above the previous best ever*** (MA or higher C levels) PIT wires.
- 100 m length multis now being made, working to translate full improvements to these conductors
- An MgB₂ layer growth mechanism was proposed for diffusion processed wires.
- Further geometry optimization and anisotropy reduction should lead to even higher J_c s and J_e s



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Summary

- High Performance Internal Magnesium Infiltration Strands are achievable with Engineering Designs that have high reacted areas inside filaments and strand fill factors
- Je performance for some of these conductors is substantially higher than PIT, and multifilamentary versions are better than PIT
- The improvements are such that they can push MgB₂ conductors into new operational regimes, to point to this we have called these more optimized Internal Magnesium Diffusion conductors, second generation
- N-values have been substantially increased, reaching above 30 for 4.2 K, 7 T, 10 K, 6 T, and 15 K-5 T.
- A wire in channel has been developed, and quench properties have been evaluated
- A coil has been wound with this WIC, and is being tested presently



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