

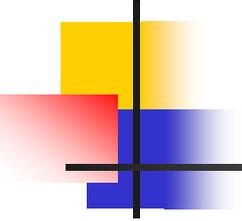
Enhanced superconducting properties of MgB_2 multifilamentary wires

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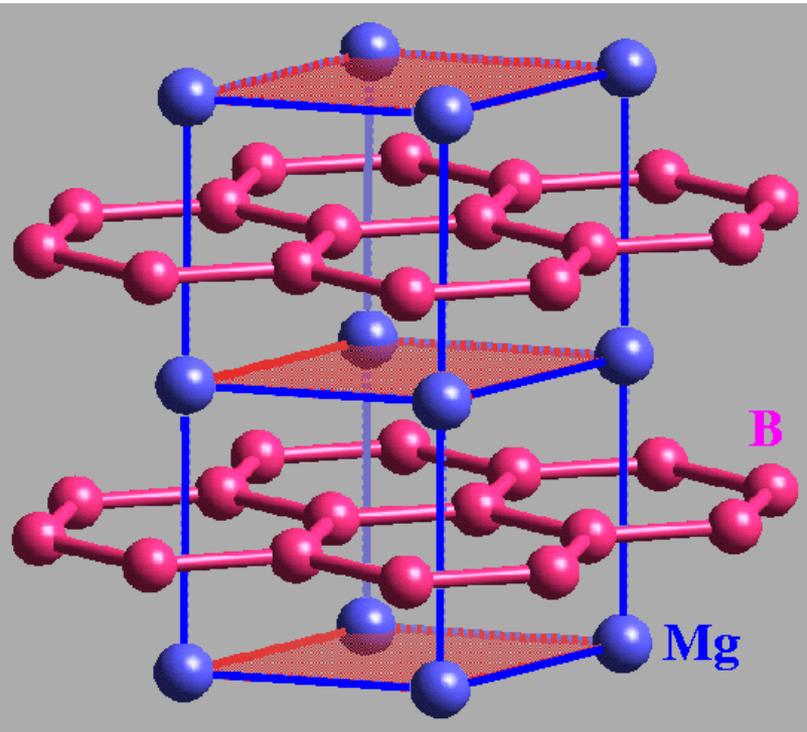
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Outline

- **Introduction**
- **High-energy ball milling to enhance J_c**
 - a) **Low purity boron powder**
 - b) **Crystalline boron powder**
- **J_c -B properties of MgB_2 multifilamentary wires**
- **Conclusions**

Superconductor-MgB₂

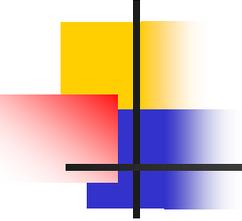


Advantage of MgB₂

- Highest T_c (~40K) among metallic superconductors
- No grain orientation required
(Easy to fabricate long tape or wire)
- Low materials cost
- Good mechanical properties
- Light weight material

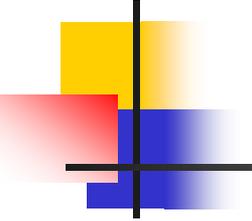
$a=3.086 \text{ \AA}$; $c=3.524 \text{ \AA}$

J. Akimitsu et al., *Nature* 410, 63 (2001).



Barrier for applications

- Many groups have already demonstrated powder-in-tube (PIT) conductors for applications.
- The key problem of MgB_2 superconductors for large-scale applications is that **the in-field J_c of multifilamentary MgB_2 wires is still lower.**



Factors influencing J_c in multifilamentary MgB_2 wires

- **Main factors influencing J_c , described in many articles:**

- Powder quality**

- Mass density of the filament**

- Reaction conditions**

- Inhomogeneity of the superconducting cores**

- **Subject of the present work**

- High quality precursor powders achieved by high-energy ball milling for multifilamentary MgB_2 wires**

How to achieve better J_c performance of MgB_2 with low purity or crystalline boron powder?

Amorphous Boron powder

- a) Very difficult to buy in the market
- b) Much more expensive

A solution: high-energy ball milling

Recently applied to wires at IEE

First tests on **low purity boron powder**, then on **crystalline boron powder**, finally for making multifilamentary MgB_2 wires.

Publications:

SuST 25 (2012) 075010; SuST 25 (2012) 035018; SuST 25 (2012) 125001

Application of high-energy ball milling on MgB₂

High-energy ball milling (HEBM) could promote the reactivity of precursor material for fabricating MgB₂ by creating clean surfaces and increasing the surface–volume ratio.

Bulk

Gumbel et al. *APL* 80 (2002) 2725

In-situ tapes

Herrmann et al. *APL* 91 (2007) 082507

Ex-situ tapes

Malagoli et al. *JAP* 104 (2008) 103908

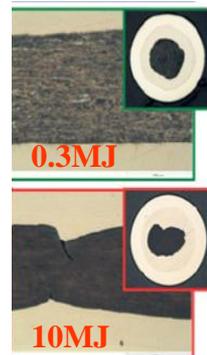
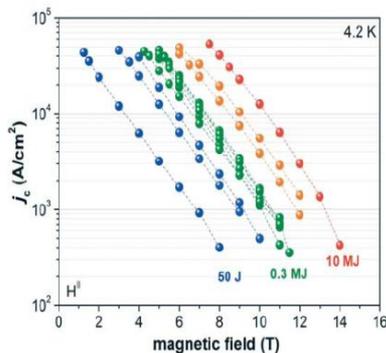
Bulk

Senkowicz et al. *SuST* 21 (2008) 035009

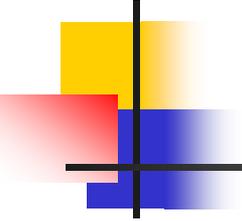
In-situ tapes

Takahashi et al. *SuST* 22 (2009) 125017

IFW



Better connectivity
Higher J_c



Low purity boron powder+ HEBM using for fabricating MgB₂ tapes

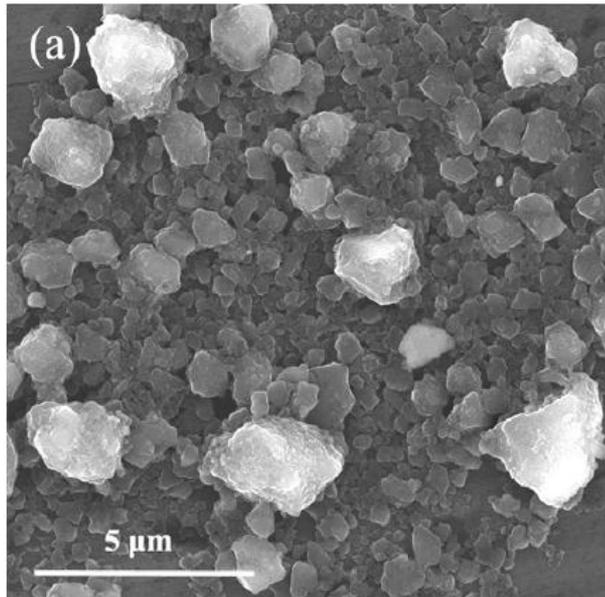
High-energy ball milling (HEBM)

- HEBM was performed in a QM-1SP4 planetary ball mill under argon atmosphere (dry milling). The milling tools were made from tungsten carbide (WC) with a cobalt (Co) binder.
- The rotational speed was 250 rpm.
- The mass ratio of ball to powder was 64.

Commercial powders of Mg (99.8%, 325 mesh, Tangshan) and Boron (96%, 1 μ m, Tangshan) were used

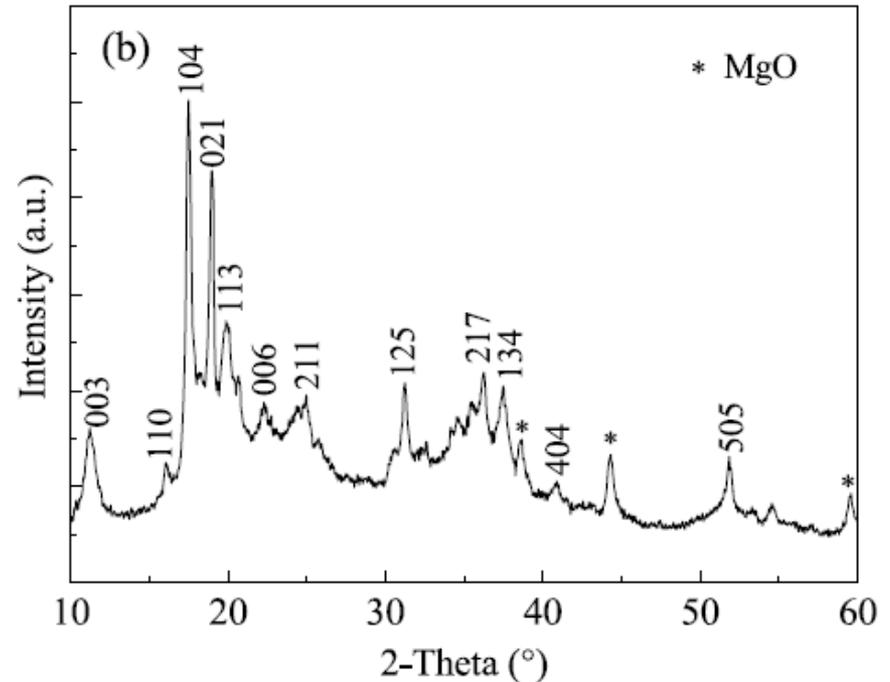
Low purity boron powder

SEM



Grain size: 1 μ m~3 μ m

XRD



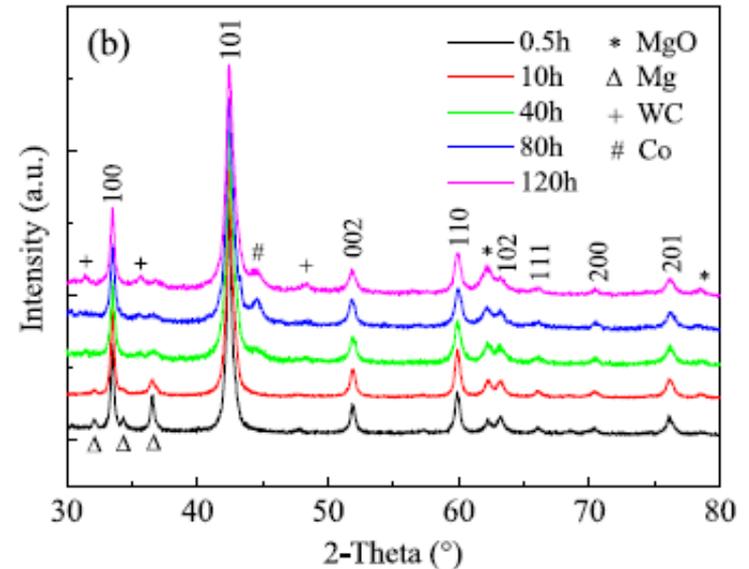
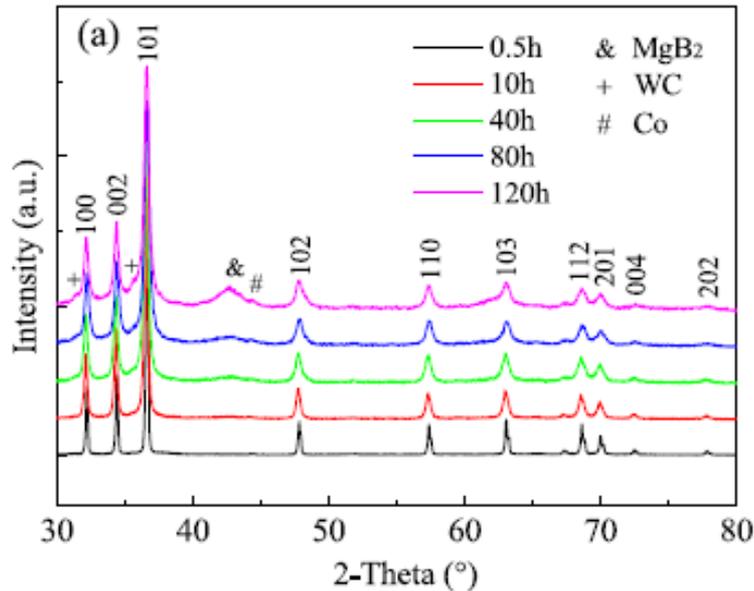
β -rhombohedral boron with small amounts of MgO

XRD patterns of ball-milled precursor powders and MgB₂ bulk samples after sintering

--Low purity boron powder

precursor powders

Bulk sample`

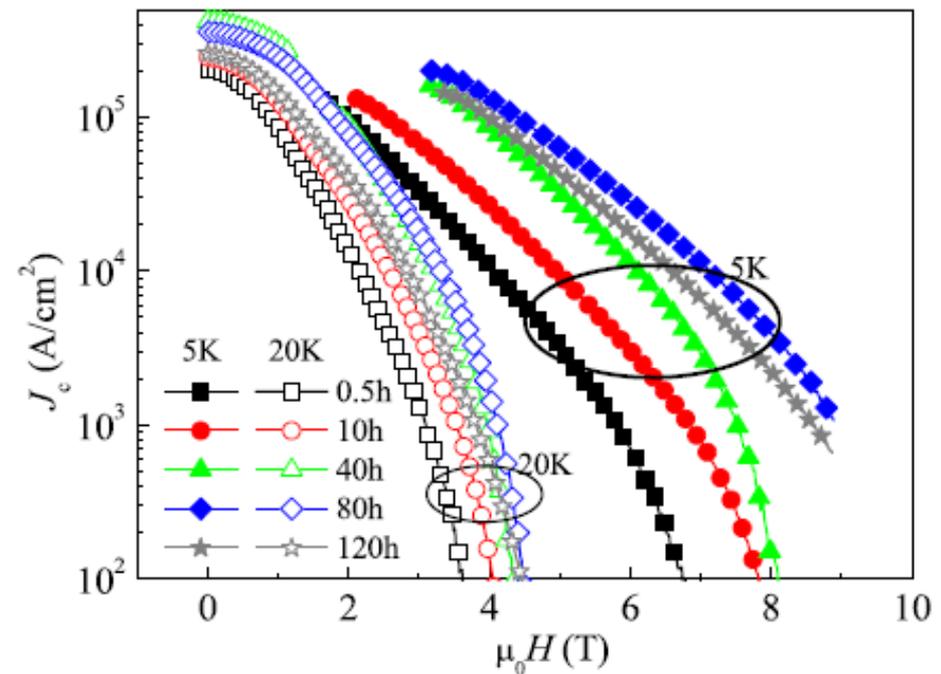
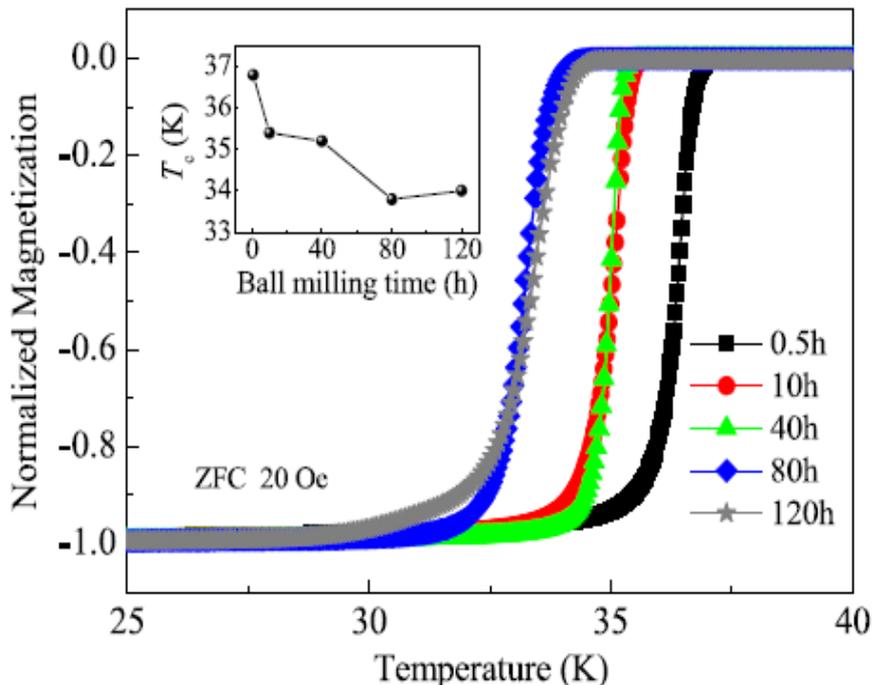


Dominant Mg diffractions are detected, MgB₂ are also visible in the precursor milled for longer than 40h

Dominant MgB₂ with small amounts of MgO and Mg

MgB₂ bulk: T_c and J_c

--Low purity boron powder

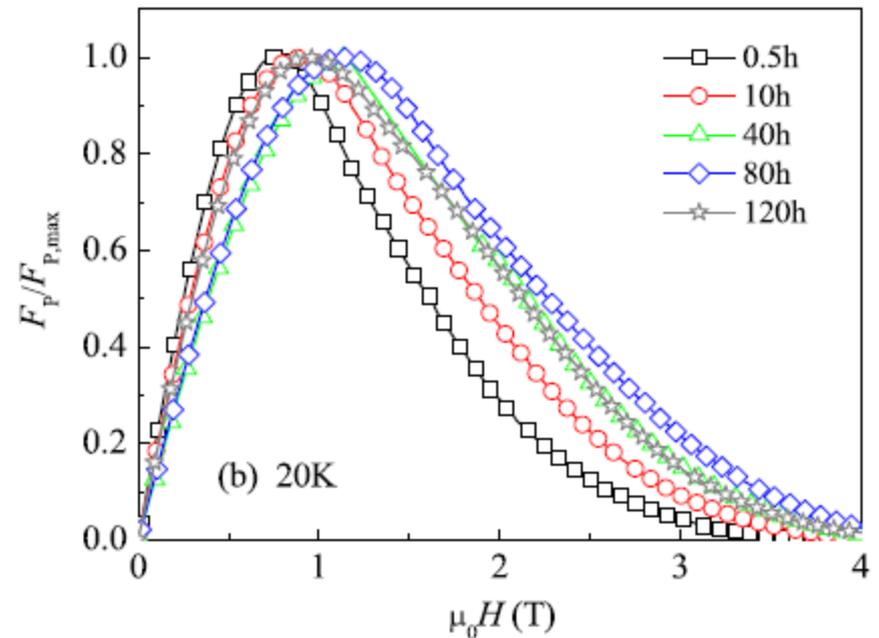
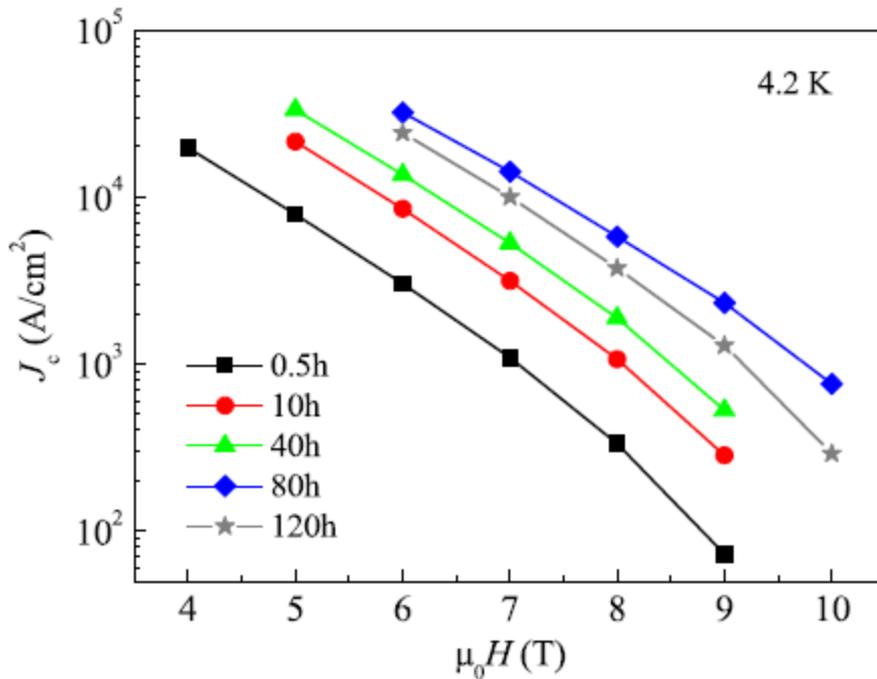


- ❑ The T_c decreases with increasing the milling time. The decrease of T_c may result from the worsening of crystallinity and the increase of crystal strain.
- ❑ The 80 h milled MgB₂ bulk sample has a J_c value of 2.8 x10⁴ A/cm² at 5 K and 6 T, 41 times larger than that of the 0.5 h sample.

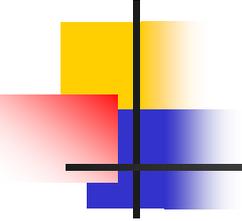
MgB₂ tapes: transport J_c increases with the milling time

MgB₂/Fe tapes sintered at 700 C

--Low purity boron powder



- Obviously, the 80 h tape has the best in-field J_c , which is consistent with the bulk samples. At 4.2 K and 9 T, the J_c of the 80 h tape reached 2320 A/cm², 32 times larger than that of the 0.5 h sample.



Crystalline boron powder + HEBM
using for fabricating MgB₂ tapes

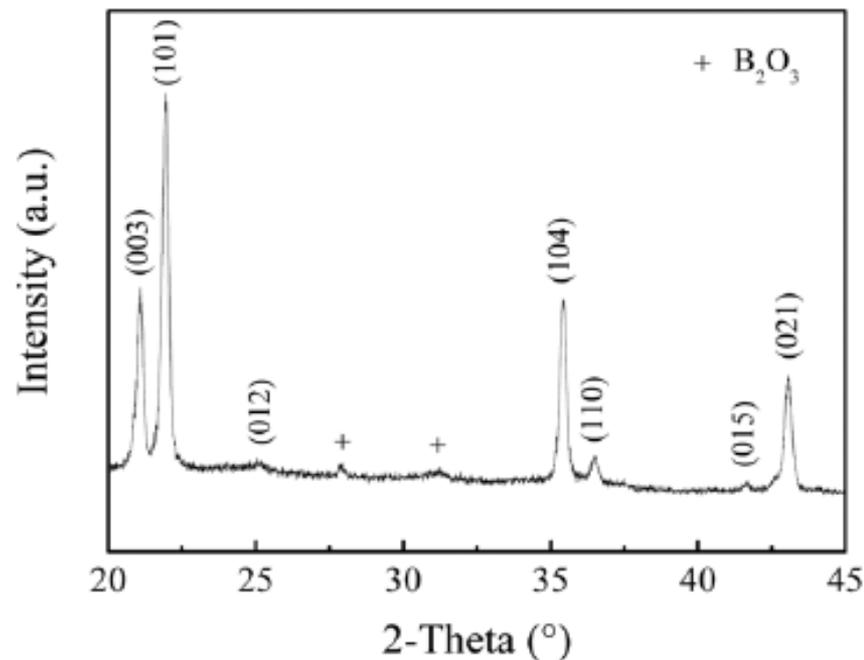
Starting materials:

Mg (99.8%, 325 mesh, Tangshan Weihao),

Boron (99.999%, 1–2 μm , crystalline, Baoding ZhongPuRuiTuo),

and carbon nanoparticle powders (20–30 nm, amorphous)

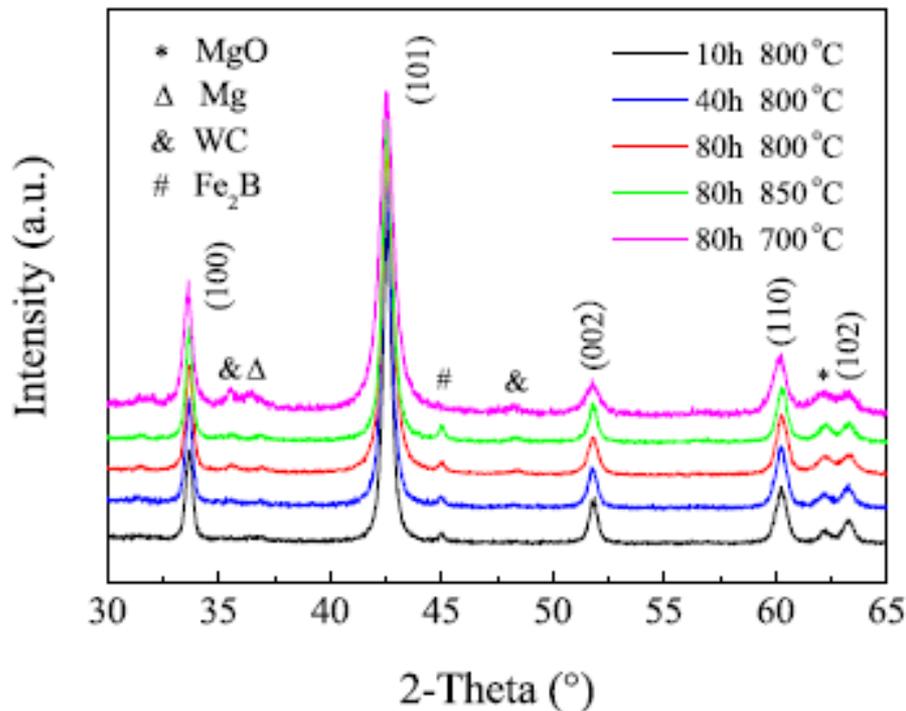
XRD of crystalline Boron powder



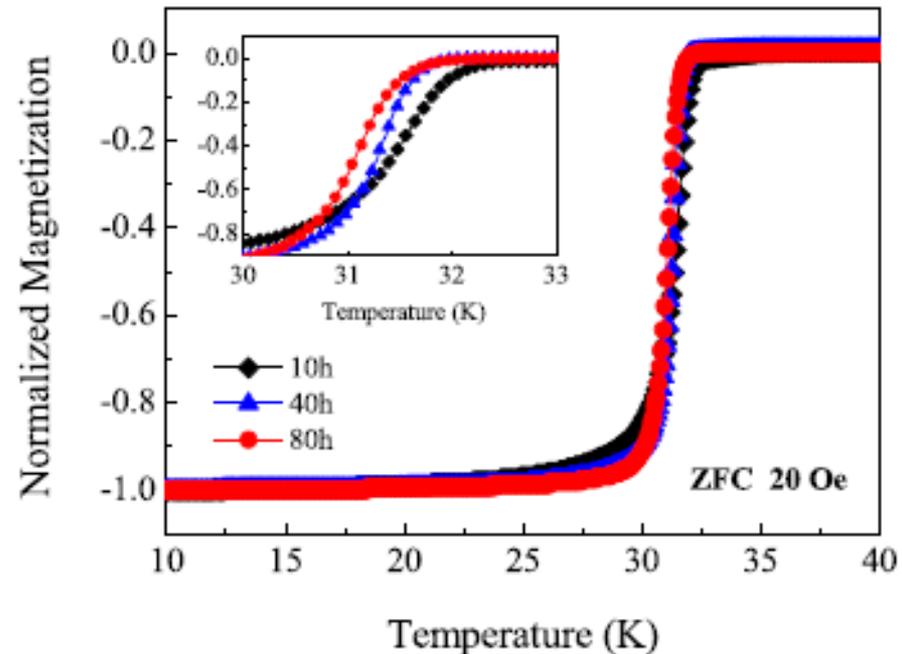
The B powder has a crystalline structure and contains small amounts of B₂O₃.

MgB₂ tapes after sintering: XRD and T_c

-- Crystalline boron powder



MgB₂ main phase with small amounts of MgO and Fe₂B.

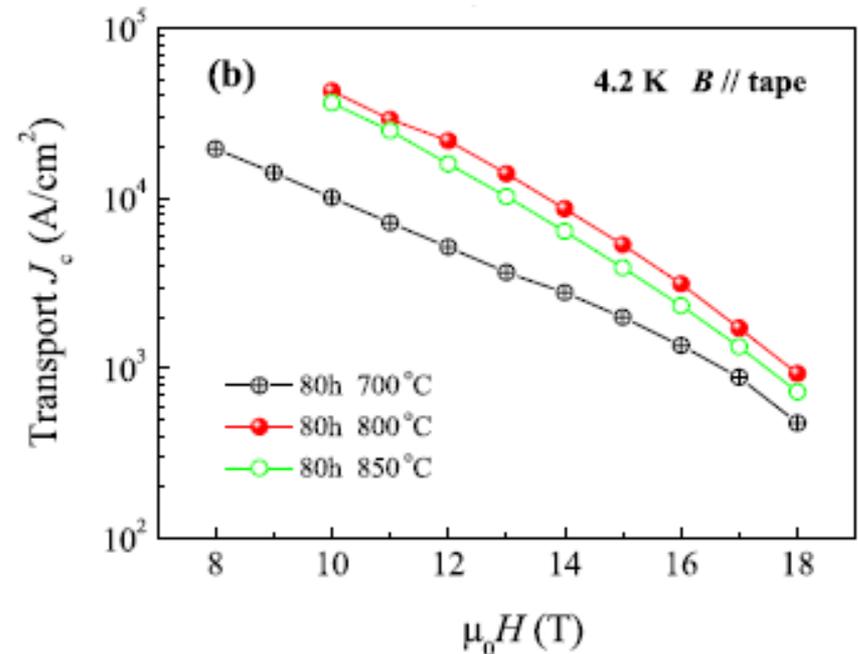
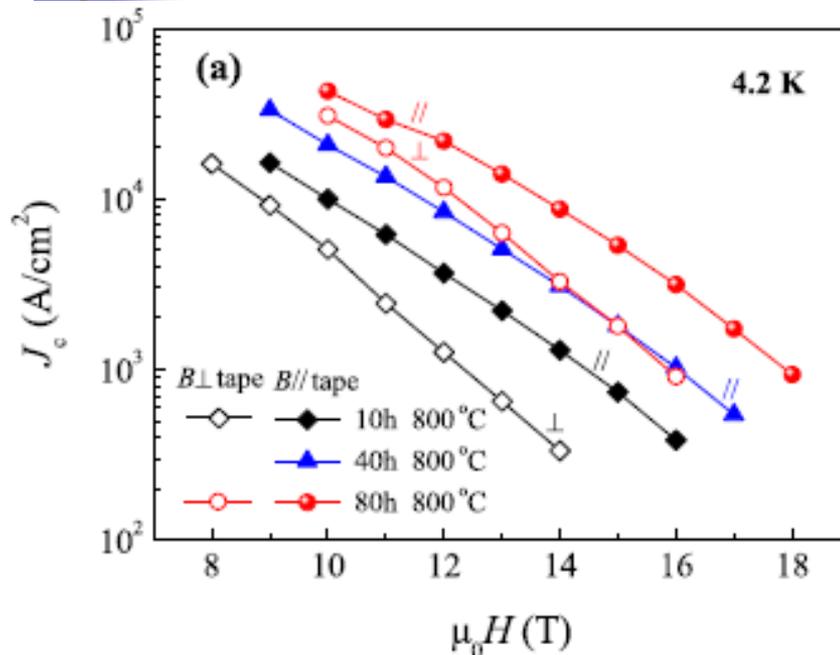


The onset T_c values slightly decrease from 32.2 to 31.9 K as milling time increases from 10 to 80 h.

In-field J_c values in MgB_2 tapes with different milling time

Fe sheath + C doping

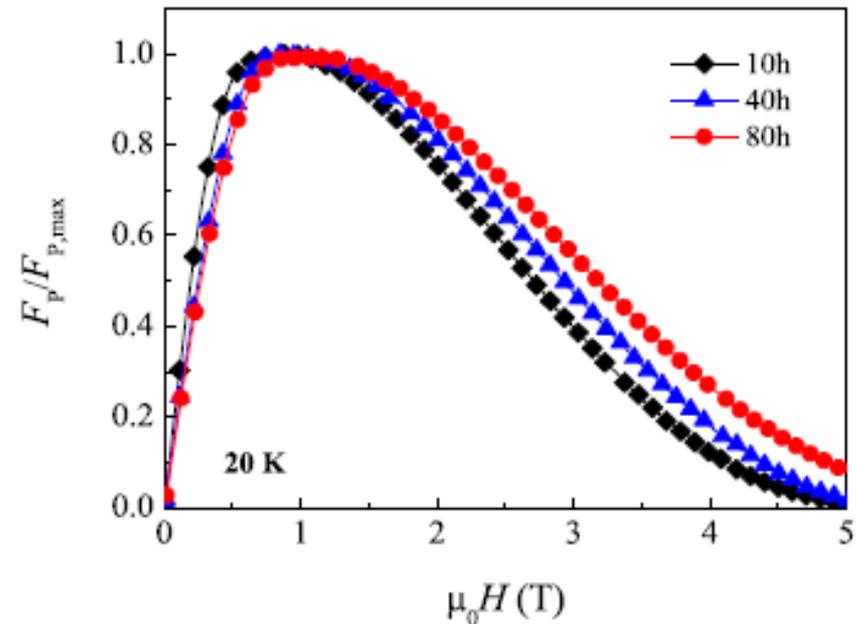
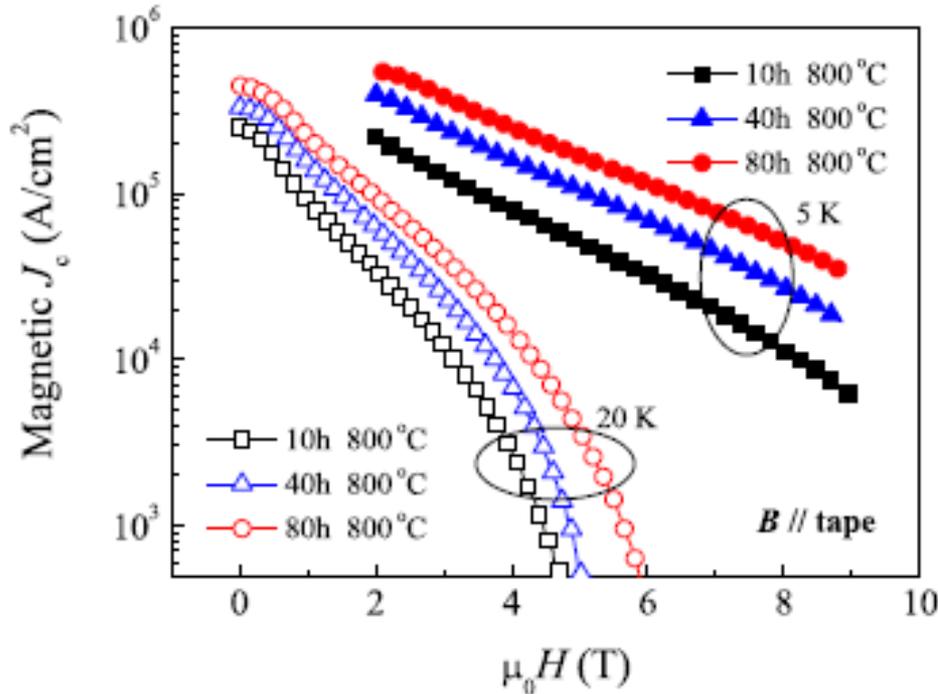
-- Crystalline boron powder



- ❑ The in-field J_c values increase with increasing the milling time up to 80 h.
- ❑ The 80 h C-doped tapes sintered at 800°C have the highest J_c of 4.3×10^4 A/cm² (4.2 K, 10 T).
- ❑ The anisotropy ratio of 10 h milled samples are 1.97 at 10 T and 3.89 at 14 T, but those of 80 h milled samples are only 1.39 and 2.65 at 10 and 14 T, respectively.

Magnetization measurement: J_c and F_p

-- Crystalline boron powder



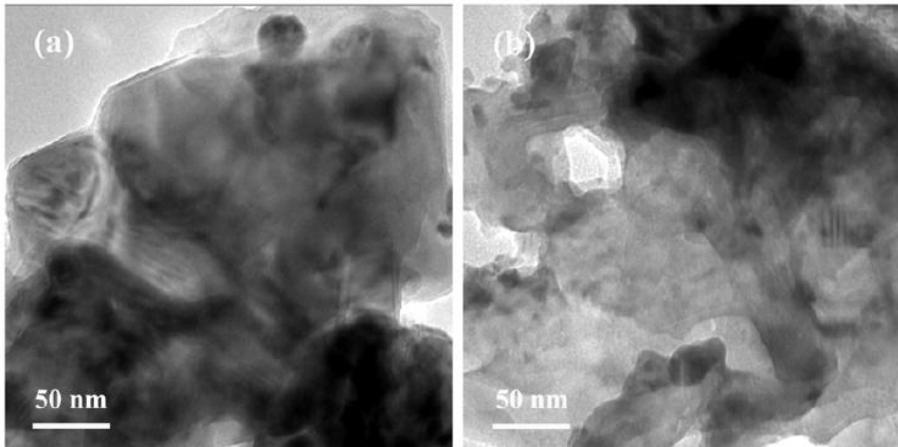
- ❑ Magnetic J_c shows an increase with increasing milling time up to 80 h at 5 and 20 K, which agrees with the transport J_c measured at 4.2 K.
- ❑ The improvement in the pinning force originates from the augmentation of grain boundaries, which is caused by the reduction in the grain size as well as the increase in defects.

TEM images of ball-milled samples

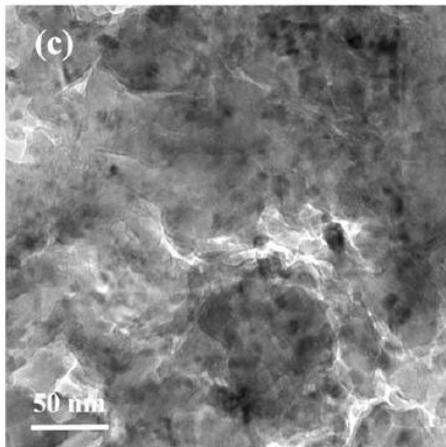
-- Crystalline boron powder

10 h

40 h



80 h

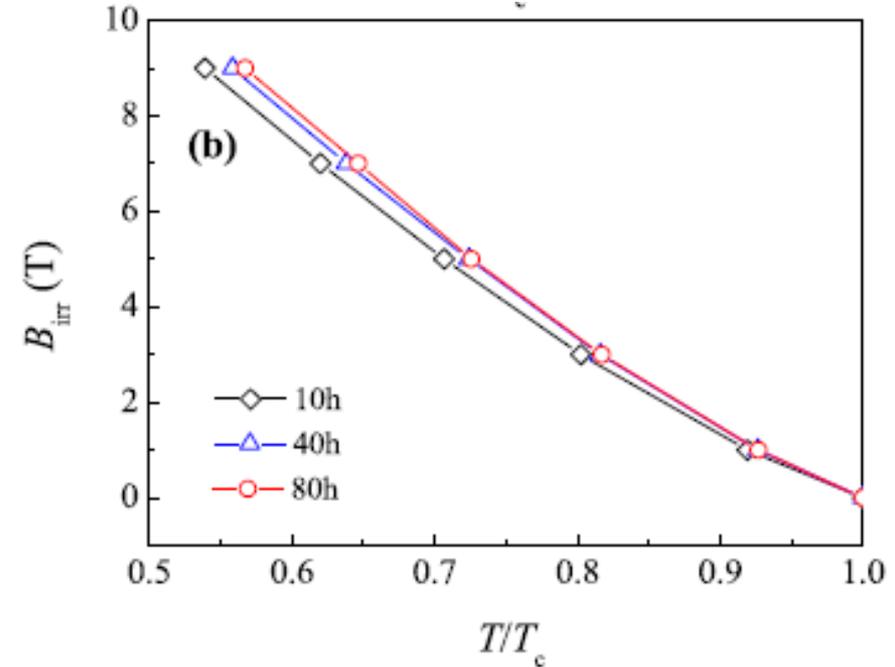
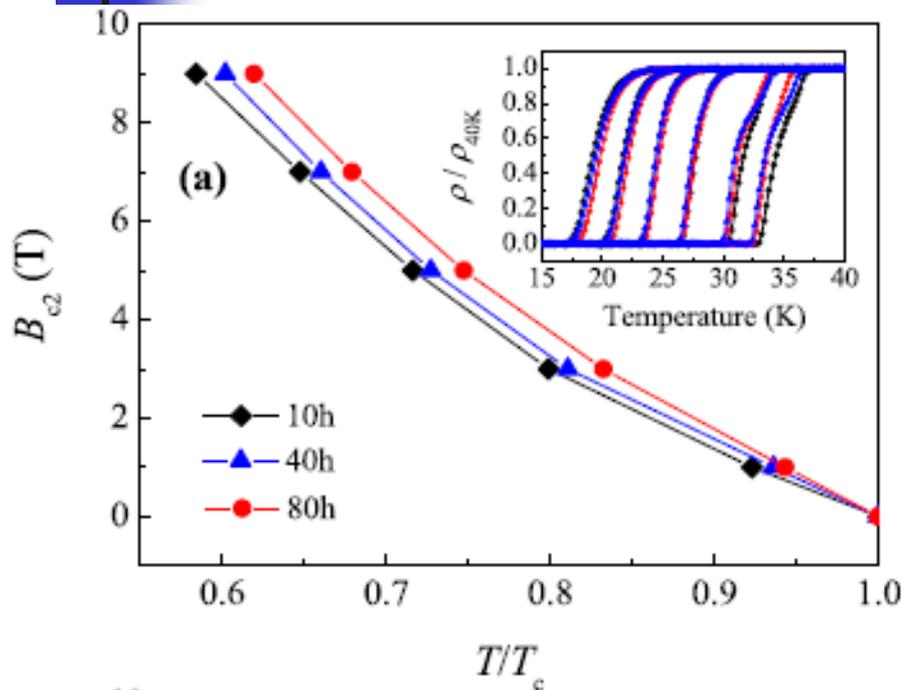


Clearly, the grain size of MgB₂ samples decreases with increasing milling time. The grain size of the 80 h sample is less than 50 nm.

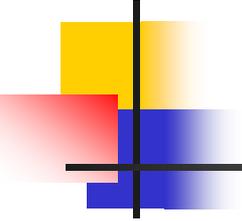
Moreover, a large number of fine precipitates less than 10 nm are scattered throughout the MgB₂ grains of the 80 h sample.

Resistance measurements: B_{c2} and B_{irr}

-- Crystalline boron powder



- The slopes of B_{c2} and B_{irr} curves rise with increasing milling time.
- The improved B_{c2} originated from a reduced coherence length due to enhanced intraband scattering as a result of the poor crystallinity and the increase in the C substitution.

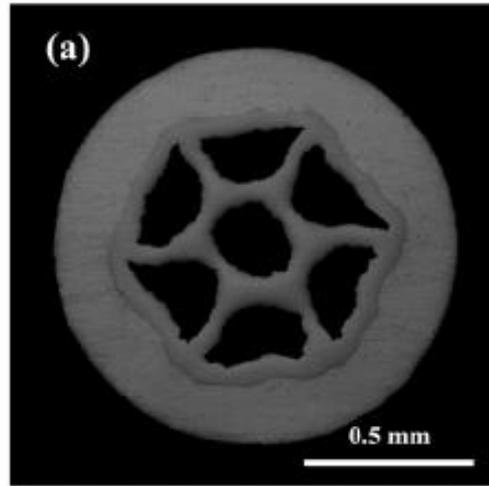


Crystalline boron powder + HEBM **using for fabricating multifilamentary wires**

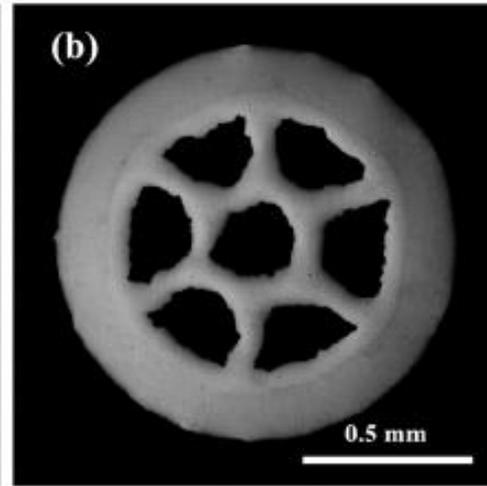
- 1) Commercial Mg (99.8%, 10 m, Tangshan) and B (99.999%, 1–2 μm , crystalline, Baoding) were weighed, while 0 and 5 wt% acetone (99.5%, Beijing) was added into the powder.**
- 2) The precursor powder was milled in a QM-1SP4 planetary ball mill (Nanjing) under argon atmosphere.**

Cross-sections of multifilamentary wires and tapes

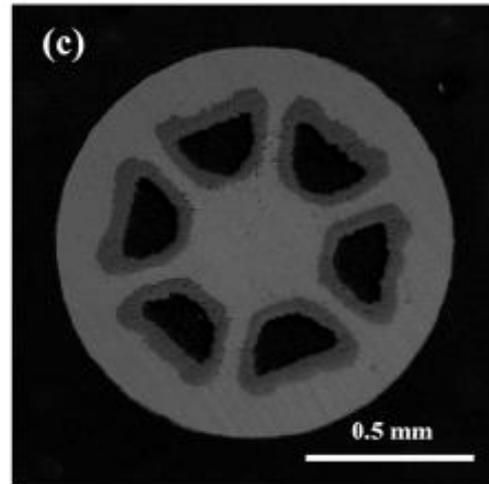
Fe/Cu



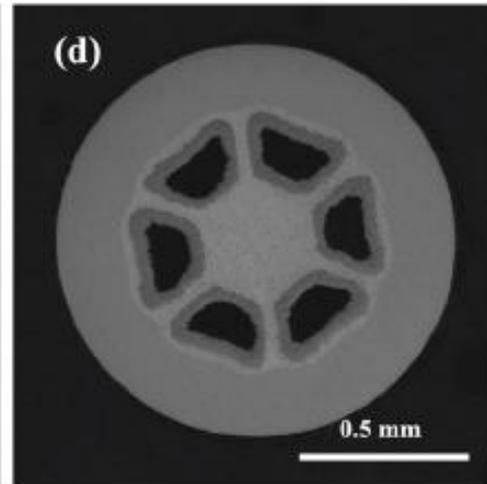
Fe/Monel



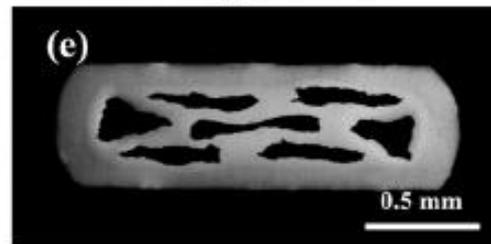
Nb/Cu



**Nb/Cu/
Monel**



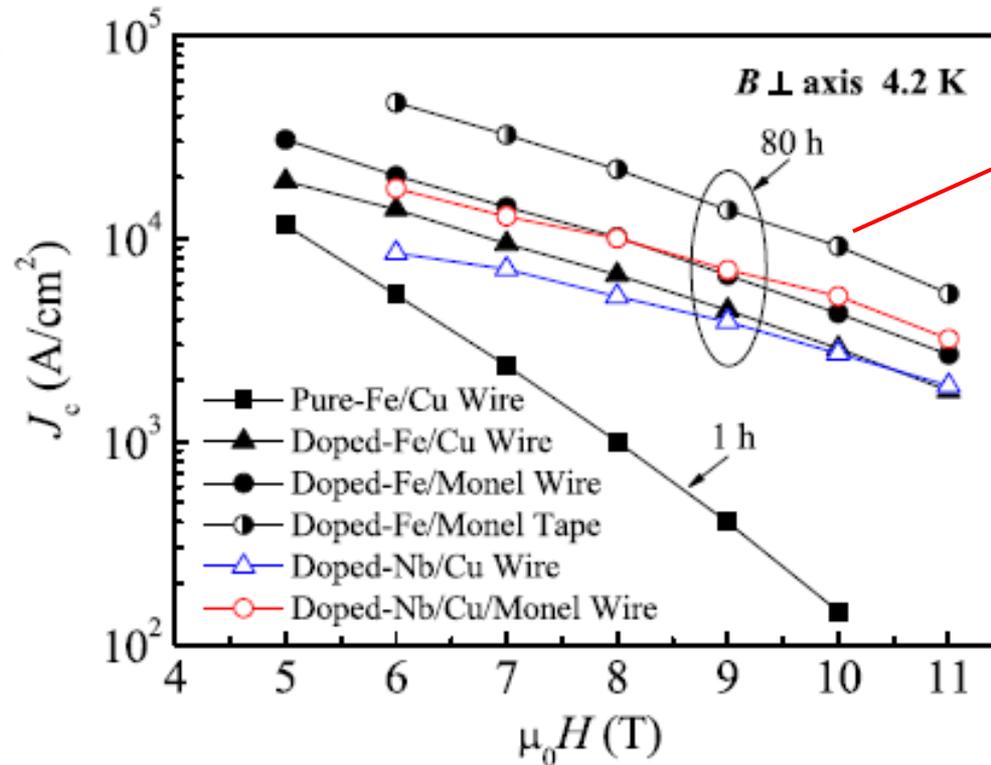
Fe/Monel



In-field J_c values in multifilamentary wires with 80 h milling time

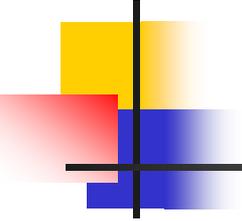
Acetone doping

The harder Monel outer sheath is beneficial to the increase of the density during deformation and better grain linkage.



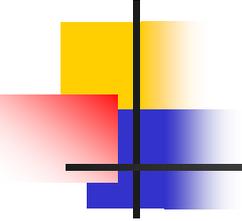
$\sim 10^4$ A/cm²

- At 4.2 K and 10 T, the J_c of doped Fe/Monel tape increased to $\sim 10^4$ A/cm², 64 times larger than that of the pure Fe/Cu wire.
- The presented J_c properties are still lower than the commercially available *in situ* wires, one reason being that the boron powders are different.



Summary

- ◆ **MgB₂ wires and tapes were fabricated by high energy ball milling using low purity boron powder and crystalline boron powder.**
- ◆ **It is found that ball milling can facilitate MgB₂ phase formation, refine the MgB₂ grains and increase the defect density. Also HEBM can reduce the anisotropy of the J_c.**
- ◆ **At 4.2 K, transport J_c reached 4.3×10^4 A/cm² at 10 T for C-doped monofilament MgB₂ tapes. The J_c of multifilamentary Fe/Monel tapes achieved $\sim 10^4$ A/cm² at 4.2 K and 10 T.**
- ◆ **The use of crystalline B in combination with a ball milling process may be an economic method to obtain the excellent properties of MgB₂ for practical applications.**



Thank you for your attention