



Enhancement of critical currents in wires of MgB_2 using different sheath materials

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Outline

- PIT method: some previous results
- Wires with different sheath materials:
 - Structural characterization
 - Superconducting characterization
- Conclusions



OBJECTIVES

- ❖ Optimization of thermal heat treatments for mono and multifilamentary wires prepared with different sheath materials (Cu, Ti, SS 316L).
- ❖ To analyze the correlation between actual defects and improving J_c



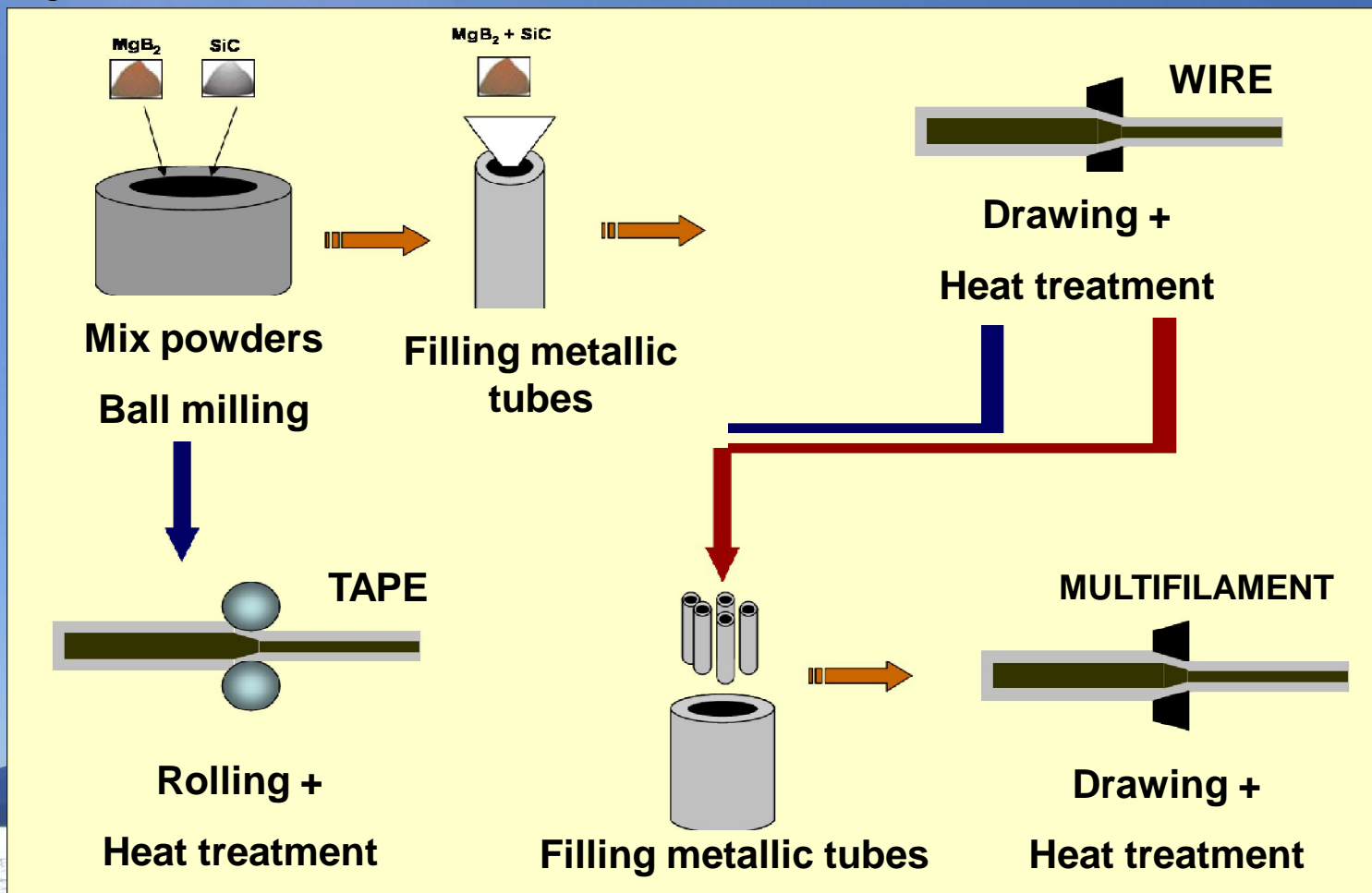
Process for PIT MgB₂ wires - tapes

Ex-situ process:

- Stainless steel as sheath material
- MgB₂ (Alfa Aesar) + 5 %at Mg excess
- MgB₂ (Alfa Aesar) + 5 %at SiC
- MgB₂ + ??

Annealings:

- Intermediate annealings at 500-900°C
- **Final annealing:** needed for removal of microcracks formed in the cold working



- Sheath
- SS,
 - Ti,
 - Ag
 - Cu

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Carbon nanotubes effects on the relaxation properties and critical current densities of *MgB₂* superconductor

G. Pasquini,^{1, a)} A. Serquis,² A. J. Moreno,¹ G. Serrano,² and L. Civale³

Journal of Applied Physics (2013) in press.



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➤ Powder in Tube (PIT) method: desired performance (J_c) already achieved in a small scale by

- ✓ Optimization of post-annealings (R. Flukiger et al. [review: Physica C 385 (2002) 286]; G. Grasso et al. [Supercond. Sci. Technol. 16 (2003) 271]; A. Serquis et al [APL 82 (2003) 1754]; A. Serquis et al [JAP 94 (2003) 4024]
- ✓ HIPing (Serquis *et al.* APL 82 (2003) 2847)
- ✓ Intermediate annealings Serano et al IEEE 15 (2005) 318
- ✓ doping with SiC (Dou *et al.* APL 81 (2002) 3419); Serquis et al IEEE 15 (2005) 3188; Serrano et al. JAP 103 (2008) 023907, Da Silva et al. IEEE Transactions on Applied Superconductivity 19 (2009) 2730-2734.

Sheath

- SS,
- Ti,
- Ag
- Cu

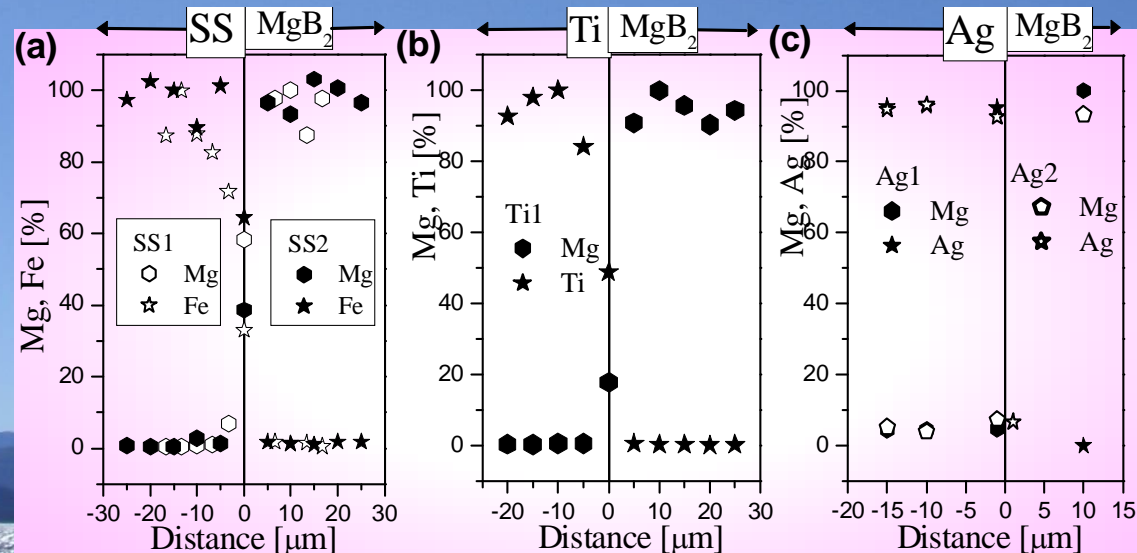
- Several intermediate heat treatments at high temperatures degrade the inter-grain critical current more than the increase in intra-grain J_c due to SiC addition
- Connectivity of MgB₂ core is a first order key parameter for obtaining good superconducting properties.

Effect of heat treatments for different sheaths

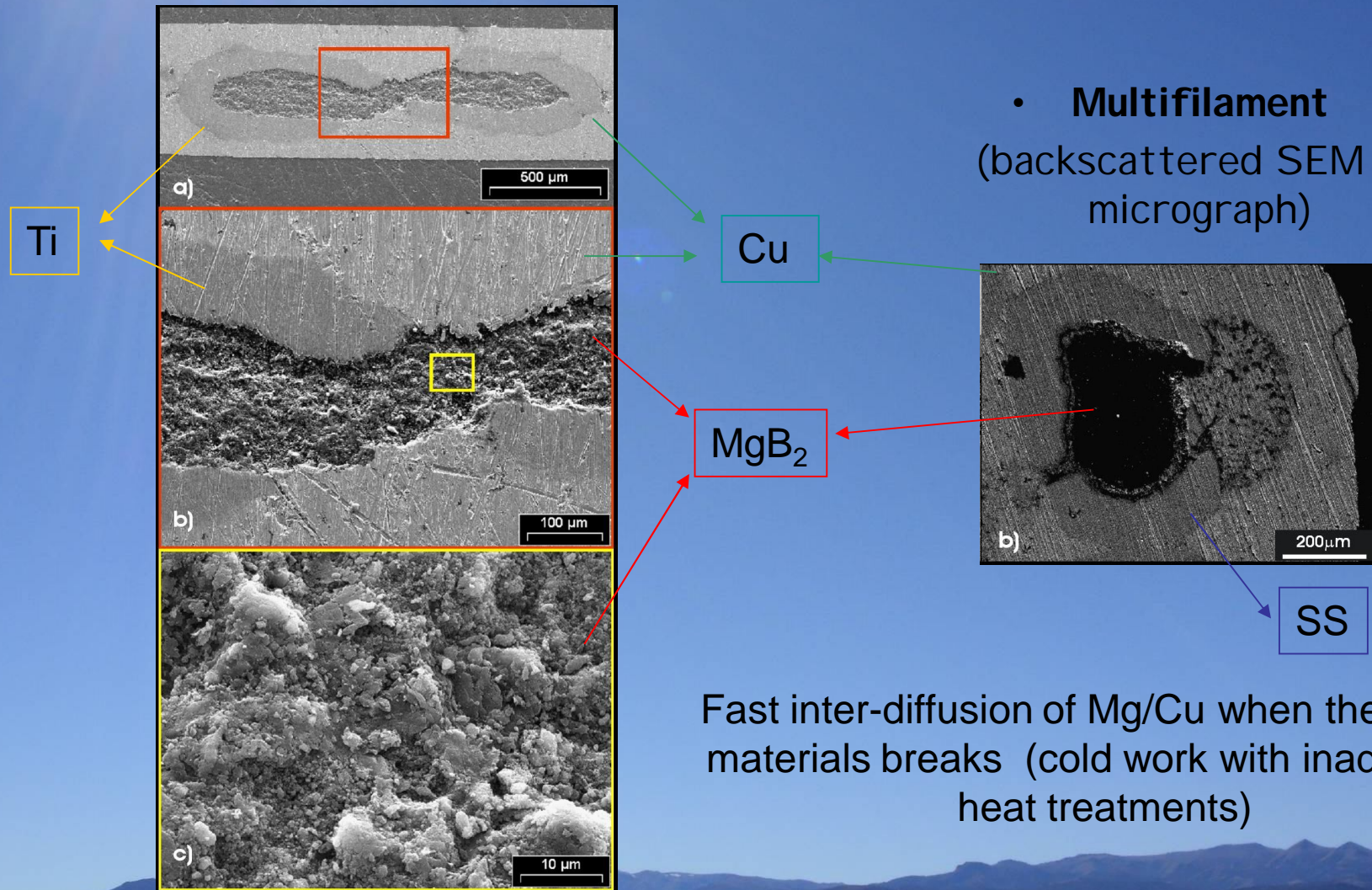
- Optimization of heat treatments suitable for each material

Sample	Sheath	Type of sample	External Size (mm)	Intermediate treatments quantity (N) and temperature (T)
SS1	stainless steel	tape	0.42 x 2.58	N = 4+ 2, T = 900°C
SS2	stainless steel	tape	0.35 x 2.85	N = 4+ 2, T = 600°C
Ti1	titanium	tape	0.35 x 2.5	N = 4, T = 600°C
Ti2	titanium/copper	tape	0.62 x 3.1	N = 1, T = 600°C
Ti3	titanium (wire) titanium/copper	multifilament tape	0.75 x 1.86	N = 4+ 3, T = 600°C
Ag1	silver	tape	0.45 x 3.7	N = 8, T = 250°C
Ag2	silver	tape	0.75 x 2.7	N = 8, T = 250°C

- Diffusion?
EDS analysis



Composite sheaths microstructure



- **Multifilament**
(backscattered SEM micrograph)

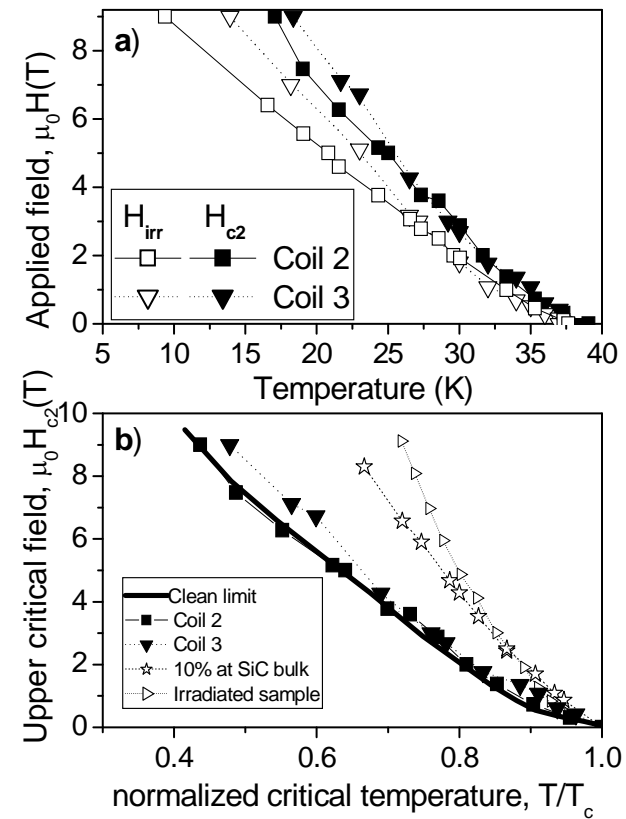
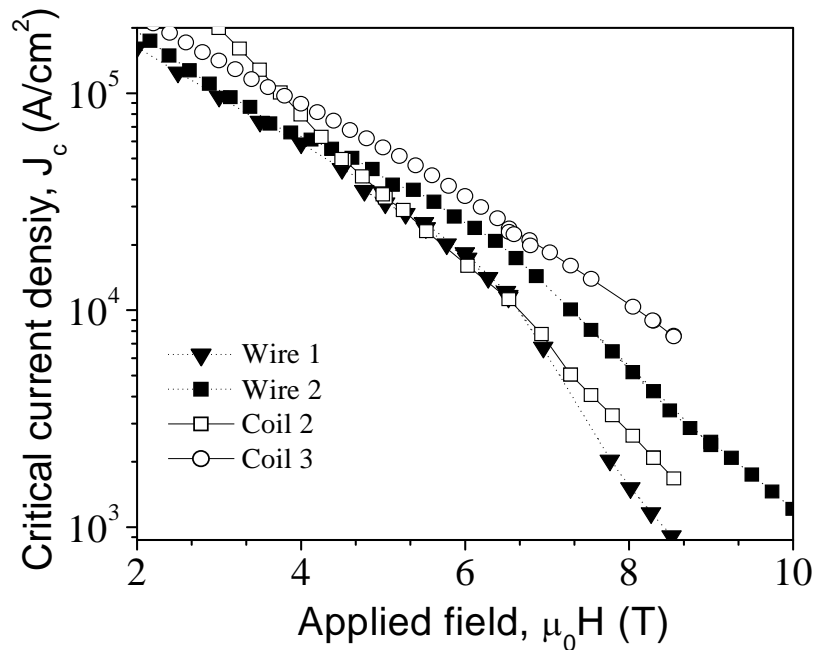
Fast inter-diffusion of Mg/Cu when the barrier materials breaks (cold work with inadequate heat treatments)

Effects of SiC doping

☑ Doping with SiC

☑ Variations in H_{c2} y H_{irr}

☑ Variations in J_C



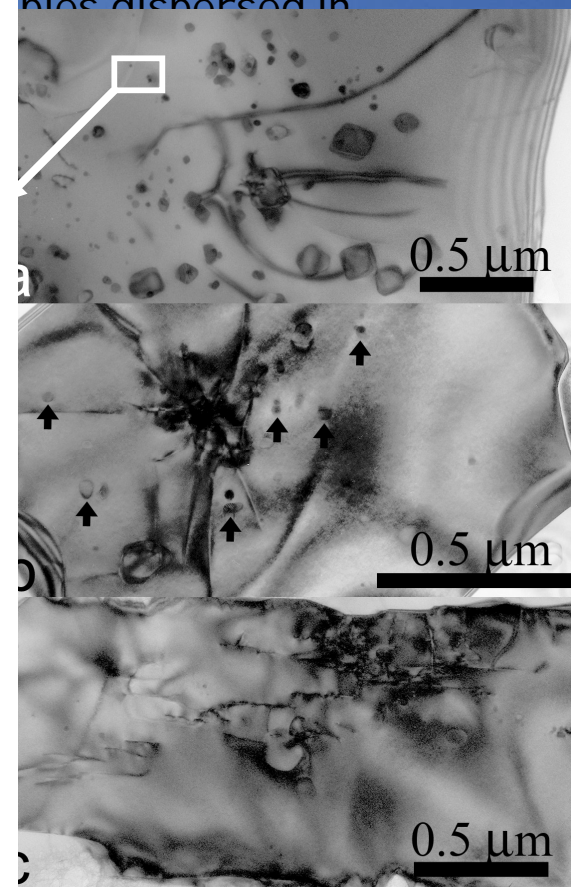
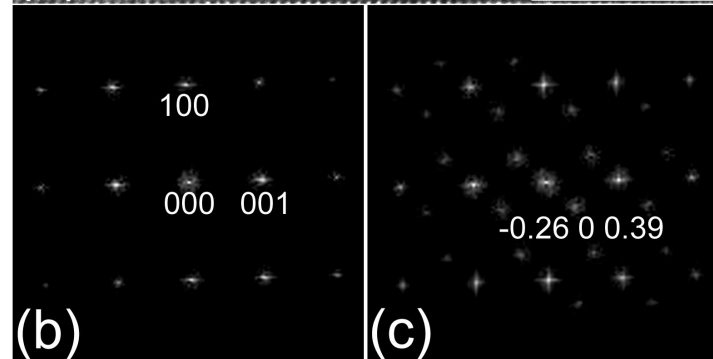
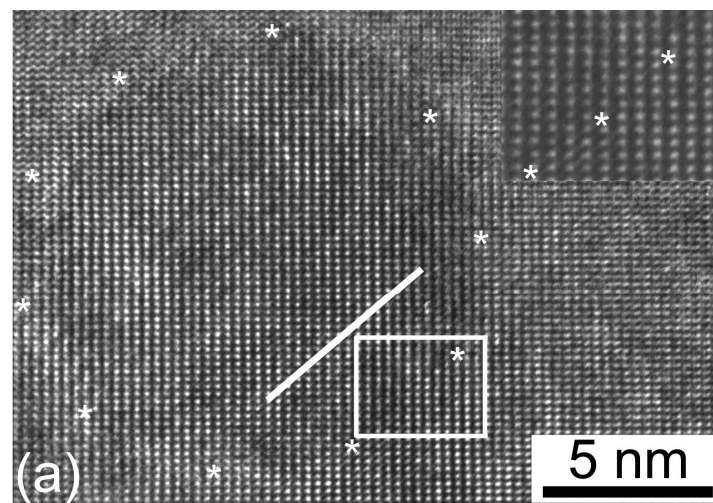
Optimization of critical currents in MgB₂ wires and coils

A. Serquis, L. Civale, D. L. Hammon, G. D. Serrano and V. F. Nesterenko

(IEEE Transactions in Applied Superconductivity 2005)

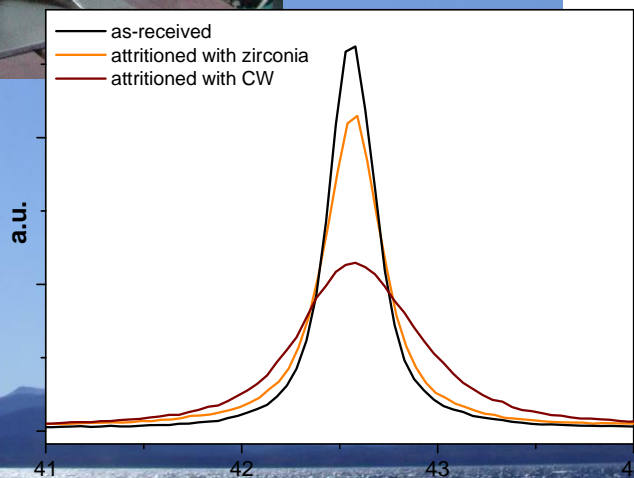
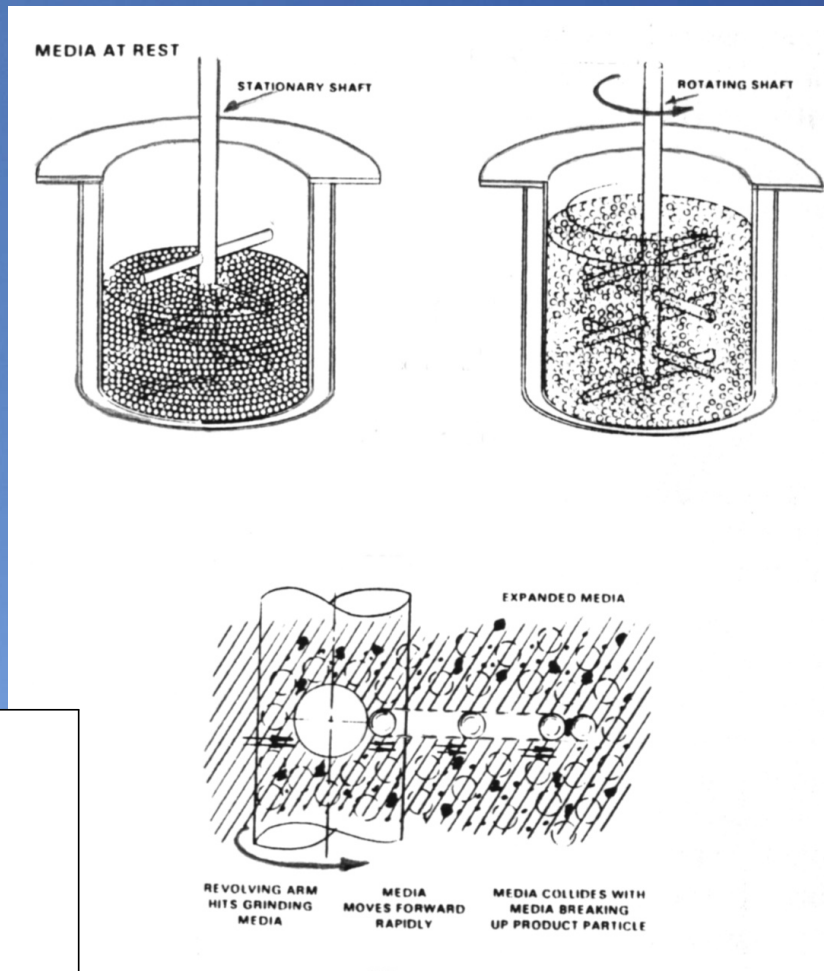
Intragranular critical currents in **bulk** samples

- Presence of $\text{Mg}(\text{B},\text{O})_2$ precipitates: X.Z. Liao *et al* APL 92, 351(2002)
- Samples sintered with different parameters

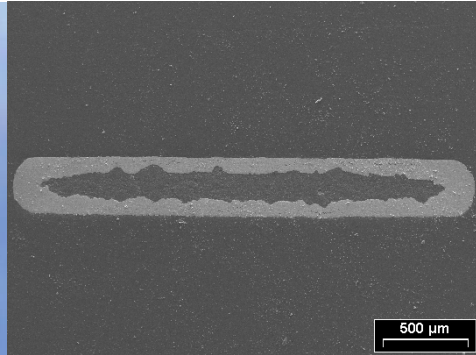


H (Oe)

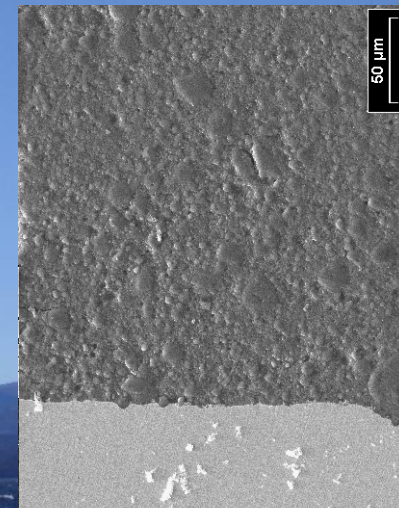
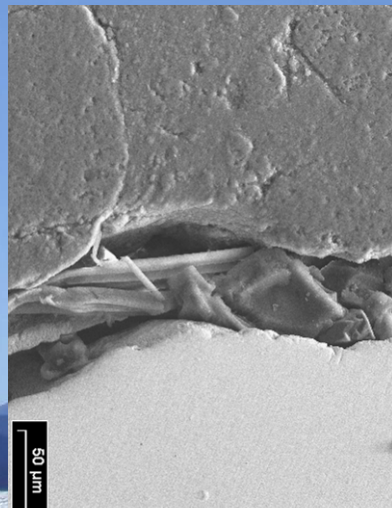
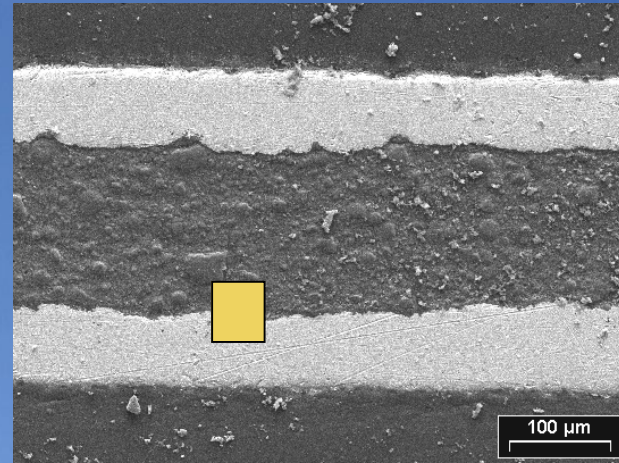
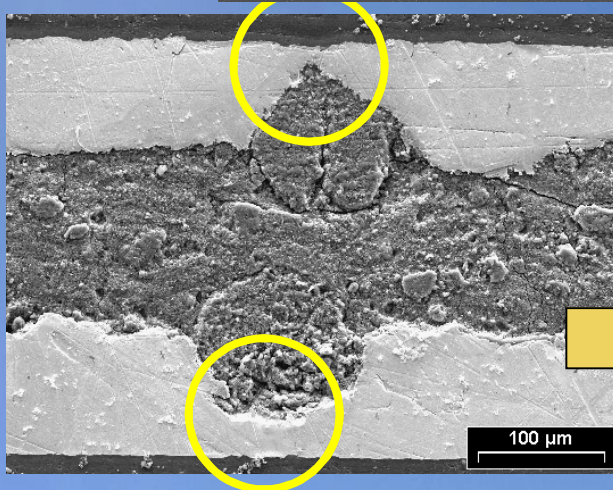
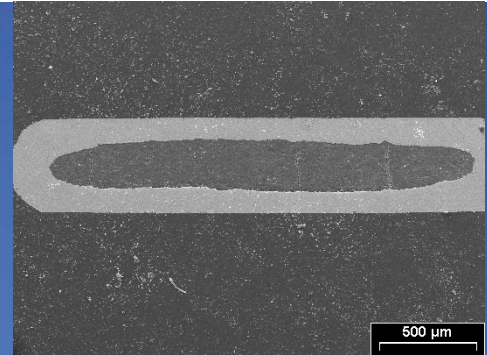
Attrition milling: reducing initial particle and crystallite size



Zr balls



CW balls



Molido con circonia

Molido con acero inoxidable

Molido con carburo de tungsteno

Powder	D [nm]	$\epsilon_g \times 10^{-3}$	T_c [K]
Z	27	0.6	38.4
SS	27	1.10	38.3
WC	17	4.25	38.2

D = tamaño de grano

ϵ_g = microdeformación (dislocaciones)

T_c = temperatura crítica

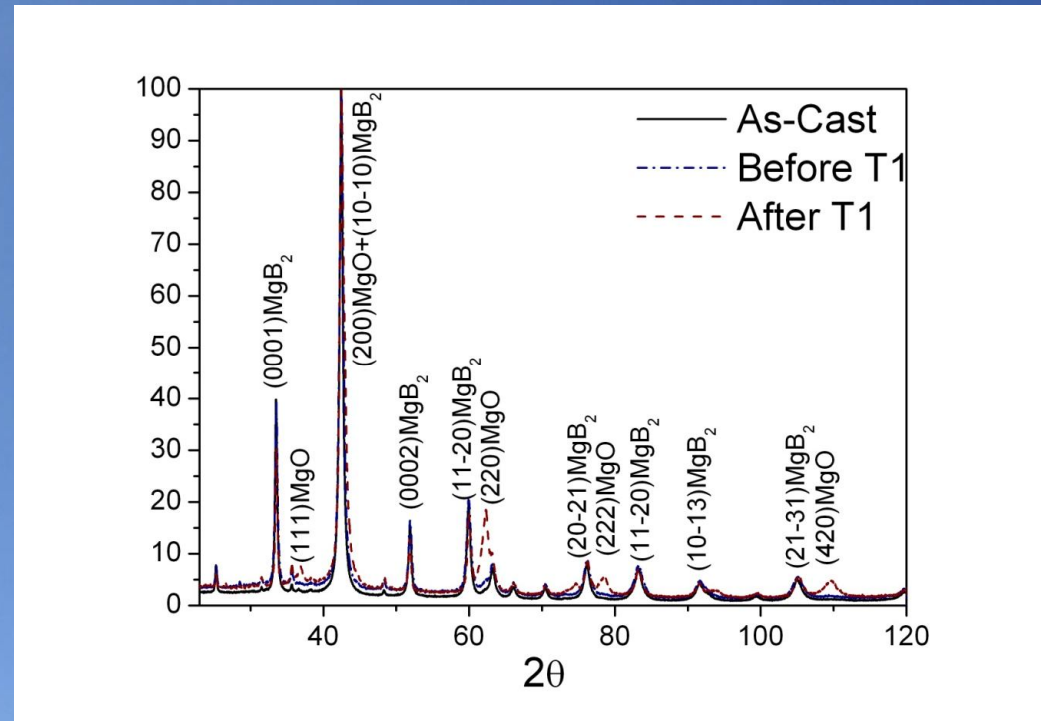
Results

Wires preparation:

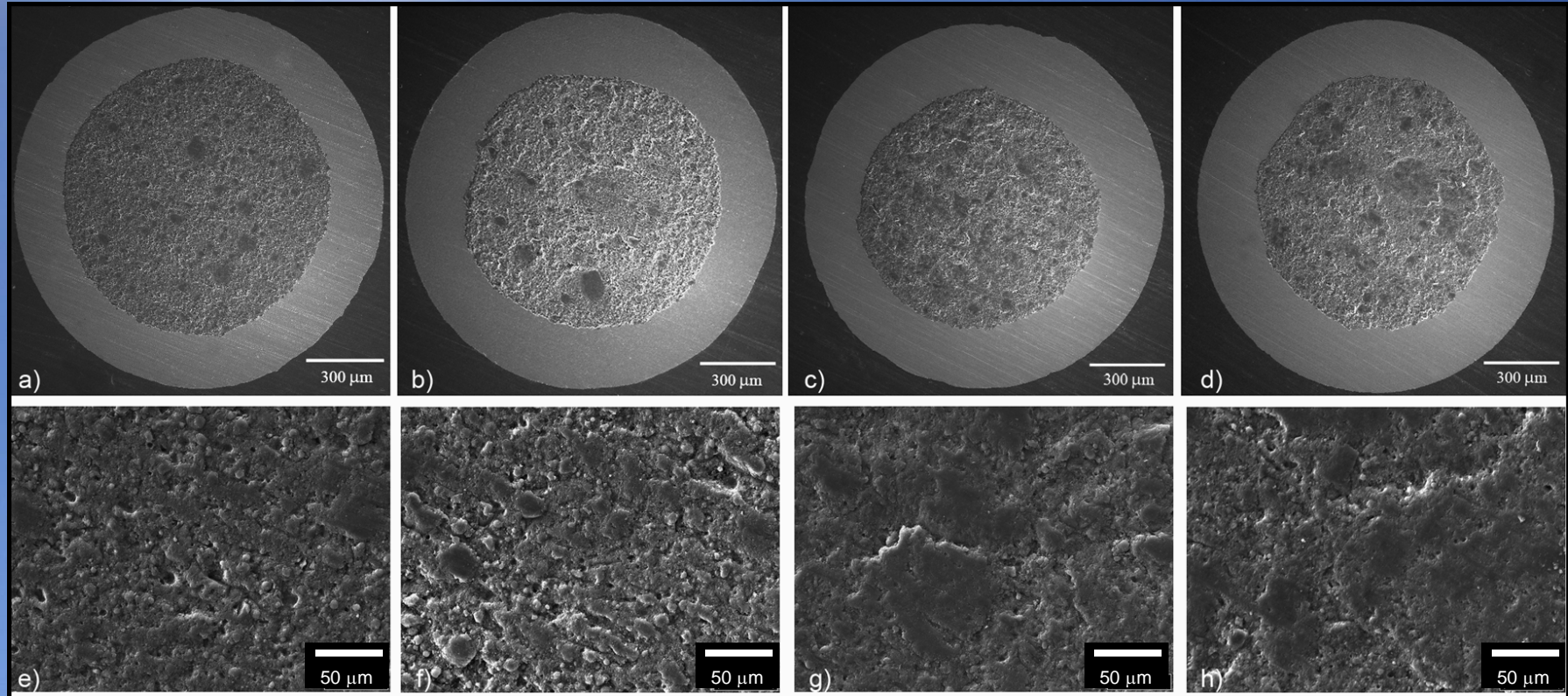
- ✓ MgB₂ milled with WC balls
- ✓ Sheath materials
- ✓ Intermediate treatments
- ✓ Final annealing

XRD Characterization:

- ✓ *Rietveld analysis*
- ✓ *Presence of MgO:*
crystal size ~7-8 nm

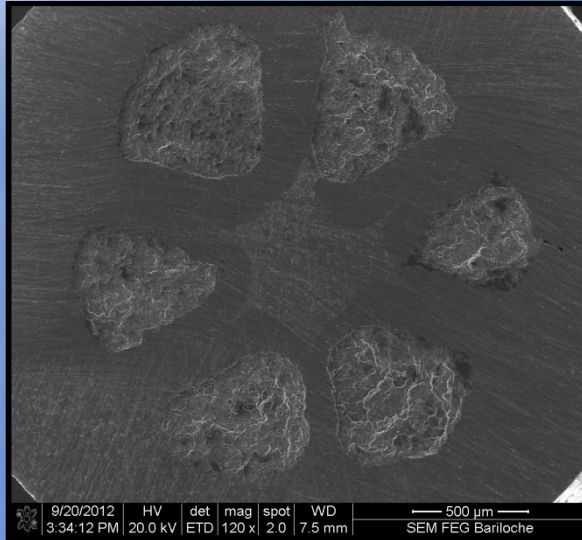


Sample	a (Å)	c (Å)	Strain XRD	Cristal Size XRD (nm)	R-Bragg
Powder	3.087	3.524	62	115	3.36
SS	3.086	3.523	70	91	2.38
SST1	3.083	3.526	45	58	3.4
CuT1	3.085	3.526	44	76	2.95

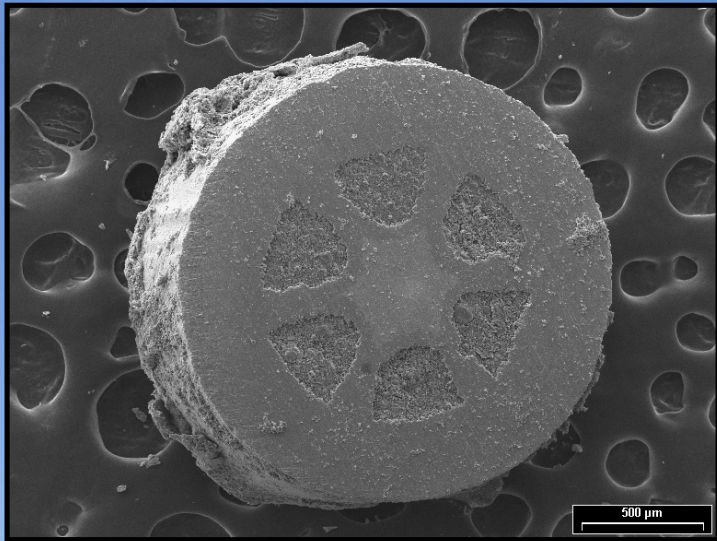


SEM of wires cross sections

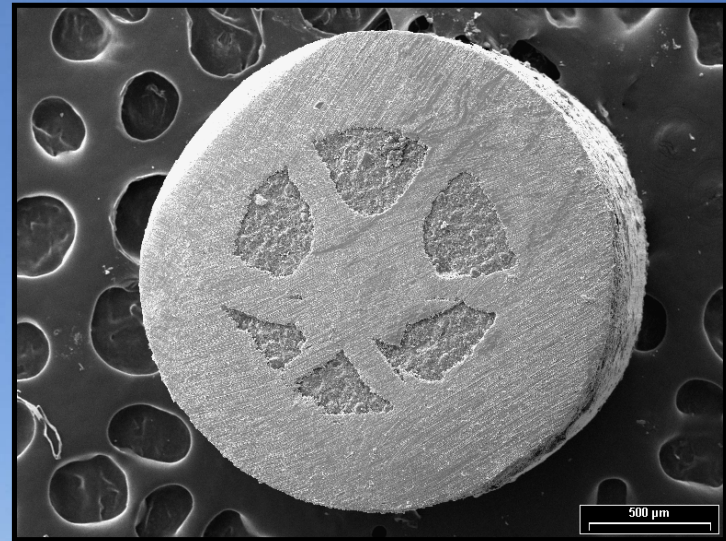
- a) Cooper
- b) Titanium
- c) Stainless Steel 316L
- d) Stainless Steel 316L (MgB2 + SiC)



SS 316L



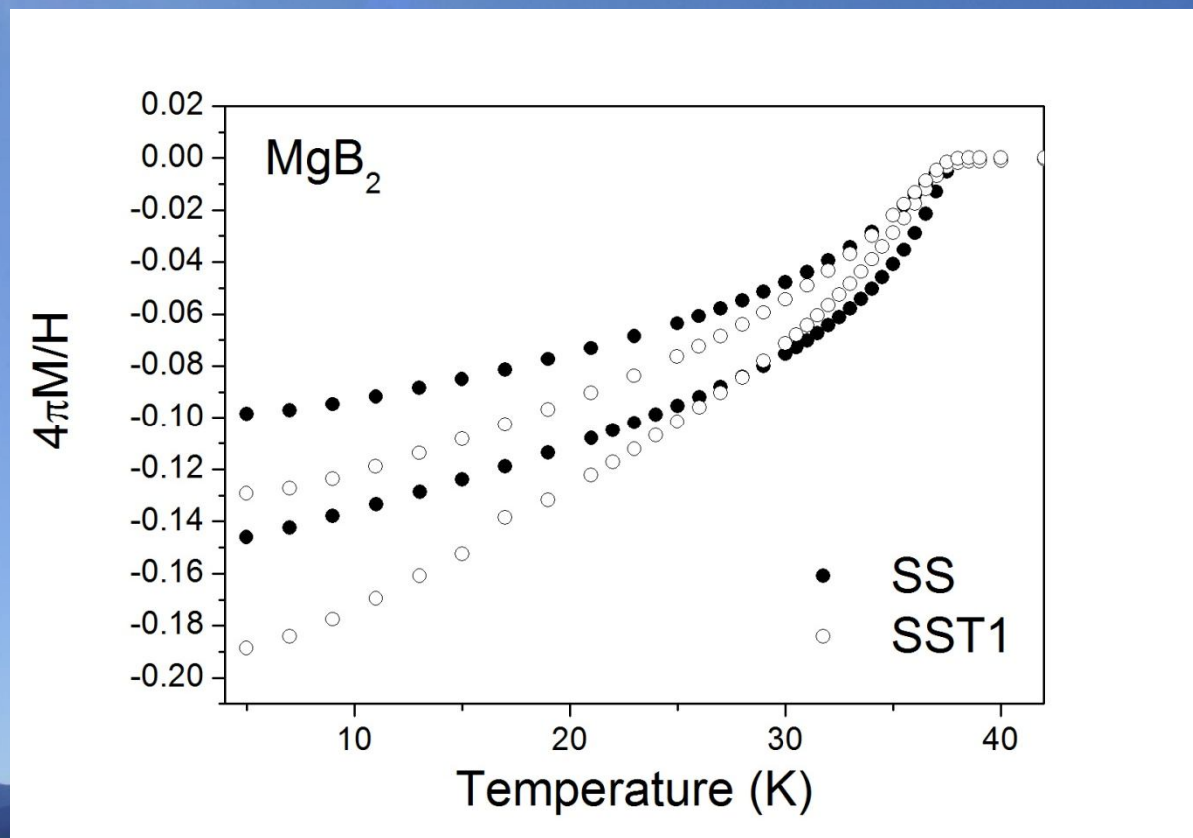
Titanium



Cooper

Superconducting properties: T_c

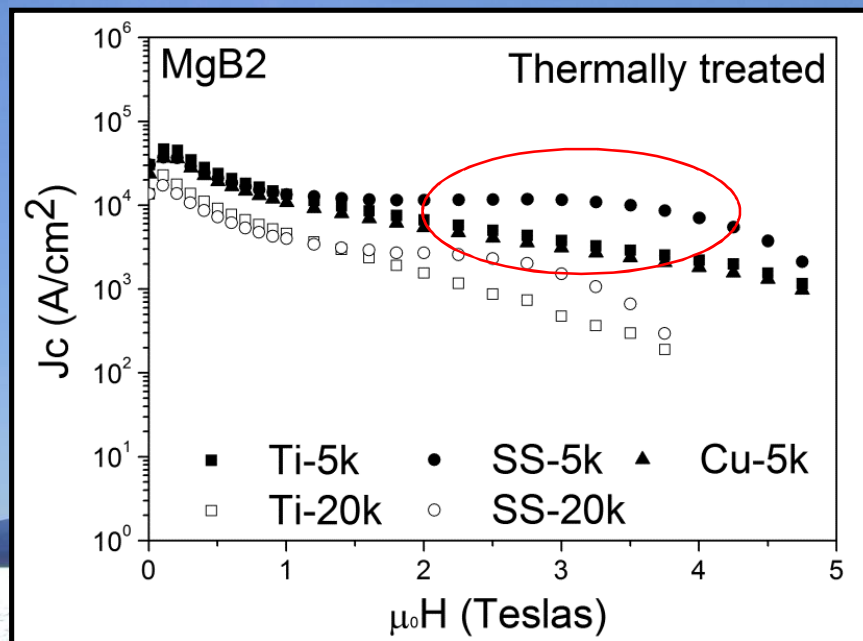
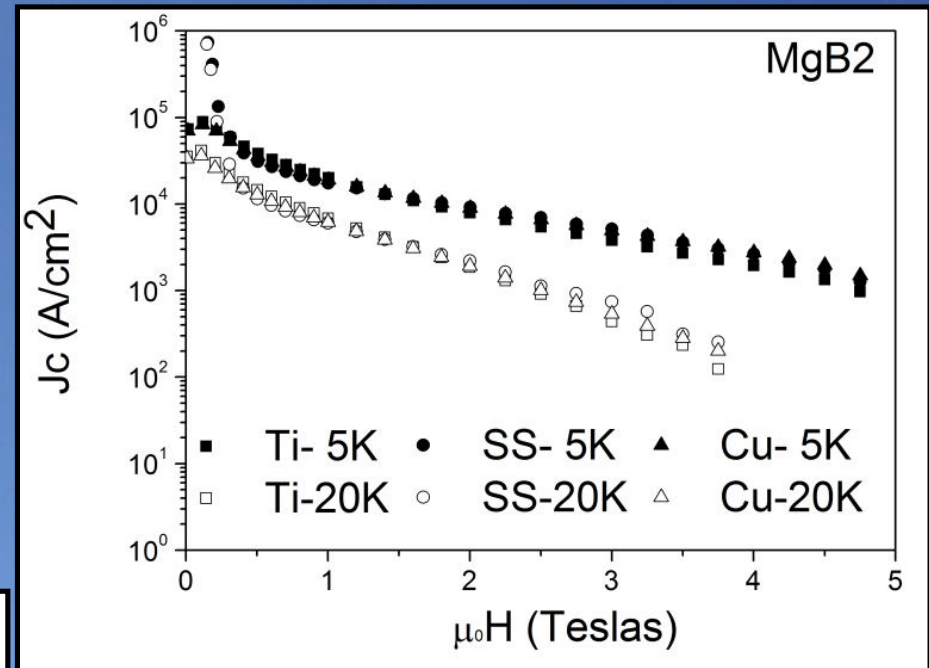
- Slightly below 38 suggests strain [A. Serquis et al Appl. Phys. Lett. 79, 4399 (2001)]



Superconducting properties: J_c

As-made wires

- ❖ Intermediate treatments: 500°C for Cu and Ti and 550°C for SS
- ❖ Slightly difference for samples before final heat treatment (even for Cu)



Wires after the final annealing

- ❖ Final annealing at 800°C during one hour under Ar atmosphere
- ❖ Particular improvement for SS: Matching field for defects?

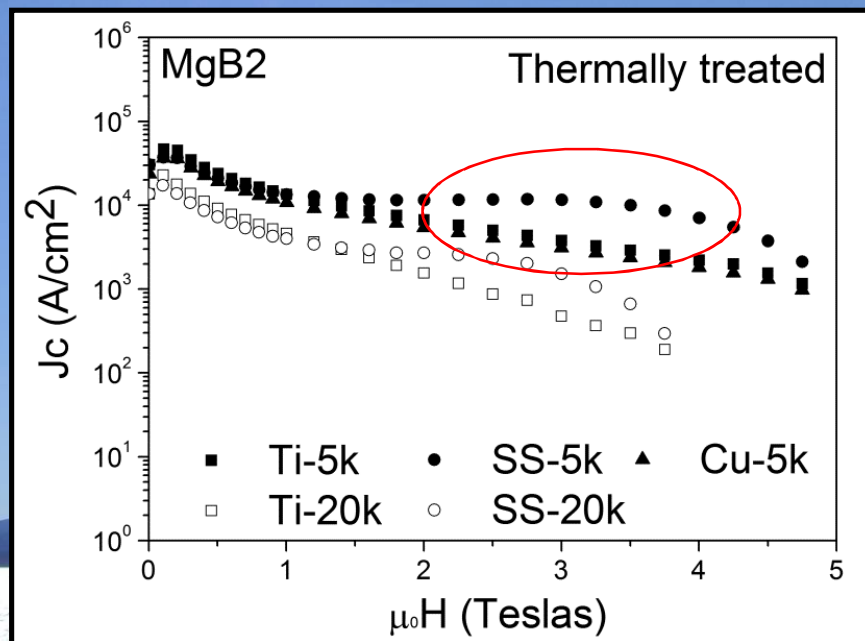
Superconducting properties: J_c

Matching field

❖ Vortex distance = $1,075 (\phi_0/B)^{1/2}$

a (2T) ~ 35 nm, a (3T) ~ 29 nm,

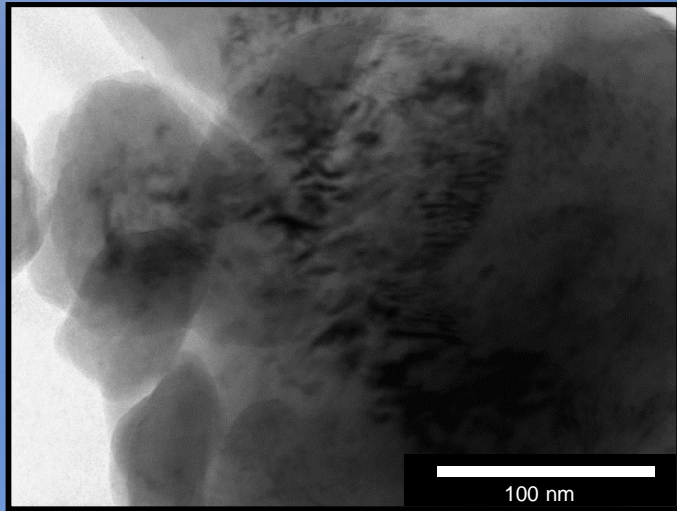
Sample	T_c [K]	J_c (5K, 3T) [A/cm ²]	J_c (20K, 3T) [A/cm ²]
Cu	37.8	0.49	0.054
Ti	37.8	0.38	0.044
SS	37.8	0.51	0.074
SSSiC	37.3	0.58	0.081
CuT1	37.9	0.31	–
TiT1	37.9	0.38	0.047
SST1	37.5	1.15×10^4	0.151



Wires after the final annealing

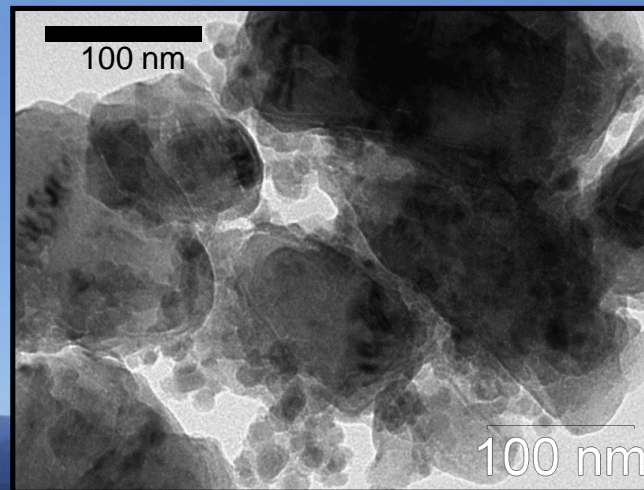
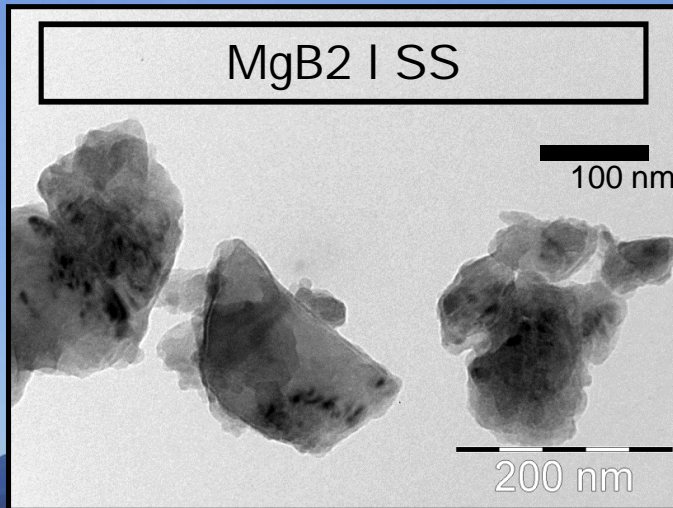
- ❖ Final annealing at 800°C during one hour under Ar atmosphere
- ❖ Particular improvement for SS: Matching field for defects?

TEM : before final TT

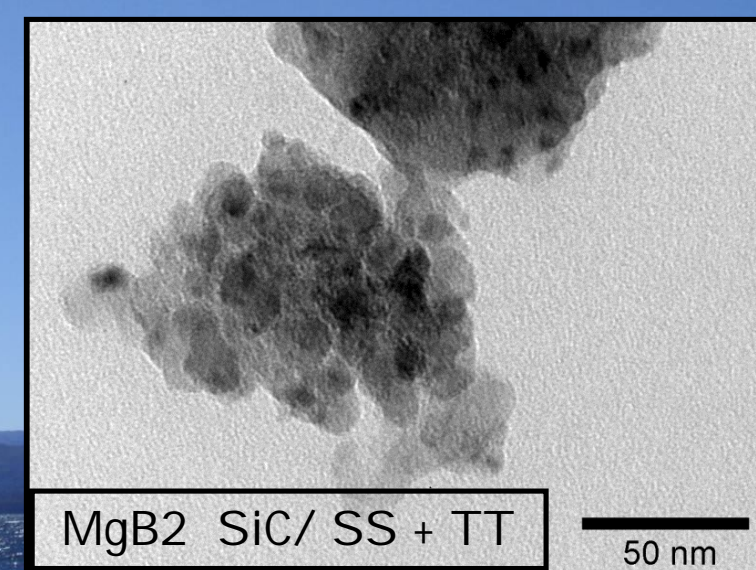
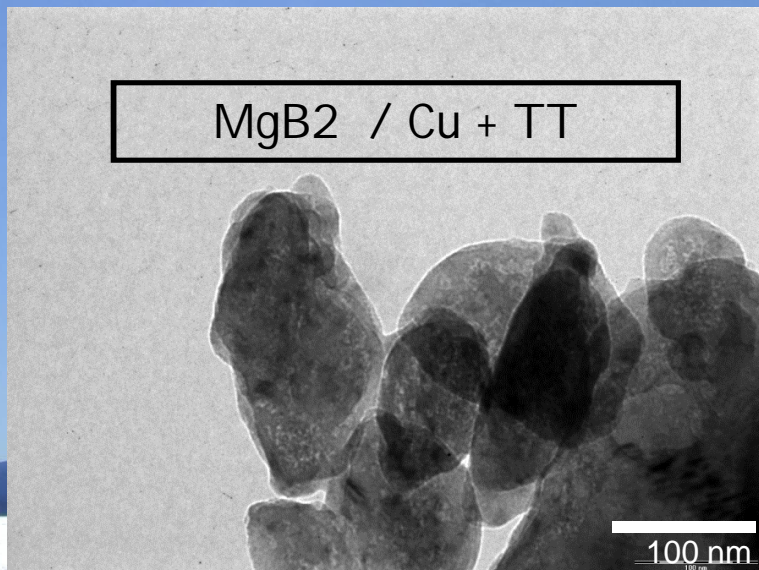
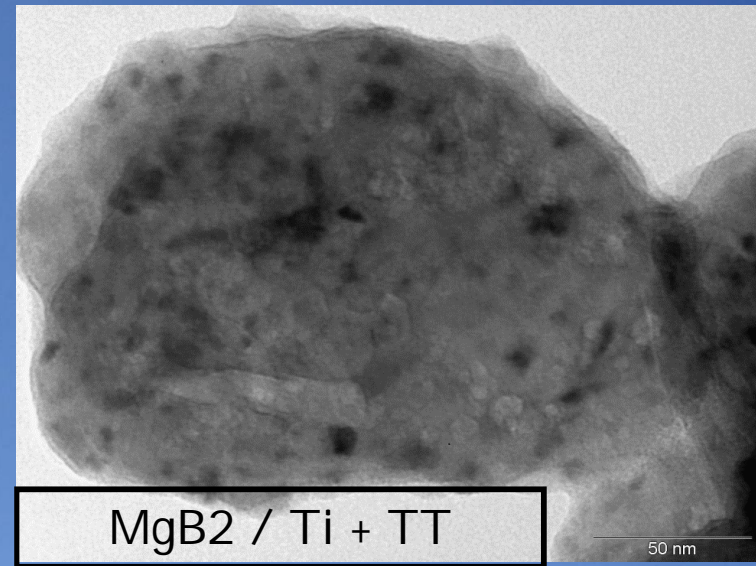
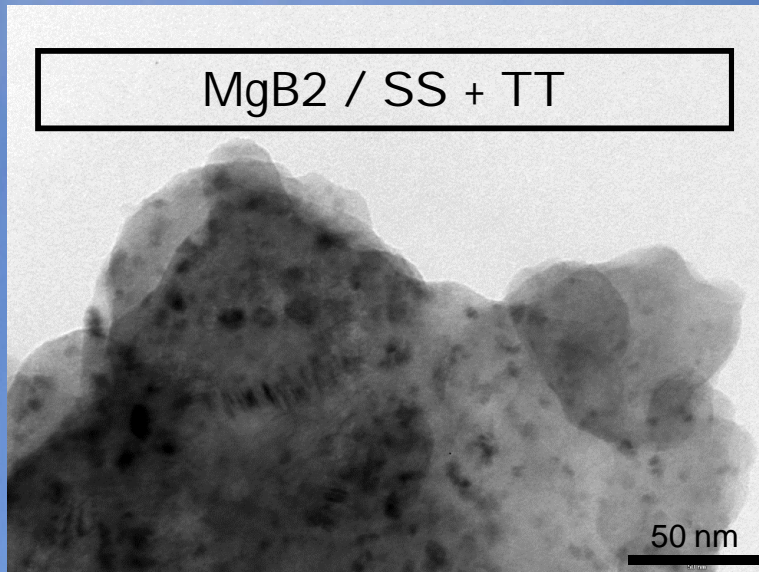


Initial MgB2 after milling

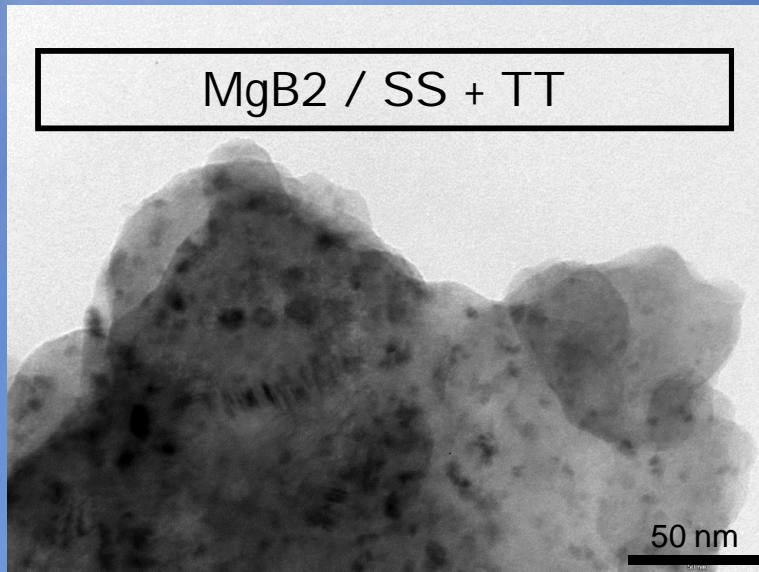
MgB2 + SiC / SS



TEM: after final TT

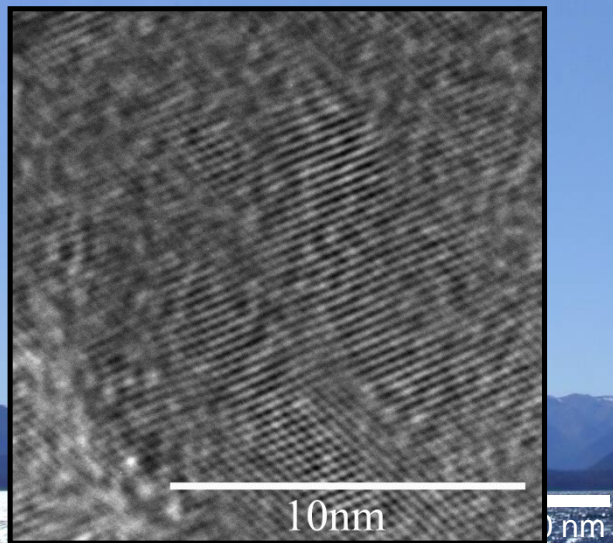


TEM: after final TT



❖ Vortex distance = $1,075 (\phi_0/B)^{1/2}$

a (2T) ~ 35 nm, a (3T) ~ 29 nm,



Mg(B, O)₂ ?

Conclusions

- ✓ PIT with different sheath materials and appropriated intermediate thermal treatment (SS, Cu, Ti)
- ✓ Final treatment at 800 C is detrimental for Cu but beneficial for SS
- ✓ Possible presence of $\text{Mg}(\text{B},\text{O})_2$ precipitates with a density that corresponds to a matching field between 2 and 3 T

Work in progress

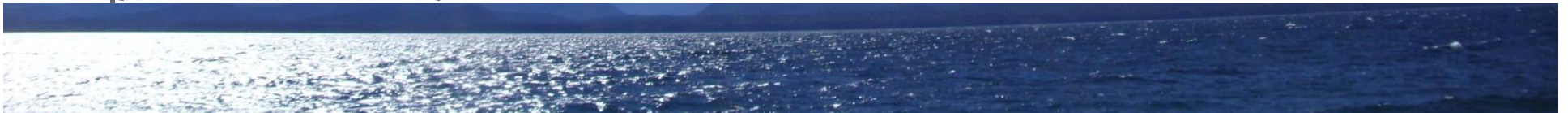
- ✓ Transport measurements
- ✓ Current transfer length
- ✓ New MgB_2 powder preparation method from boron mineral



Thanks



1 / 14 (foto lector Christian Platero)



QUESTIONS?

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