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Swiss National

Science Foundation

Outline

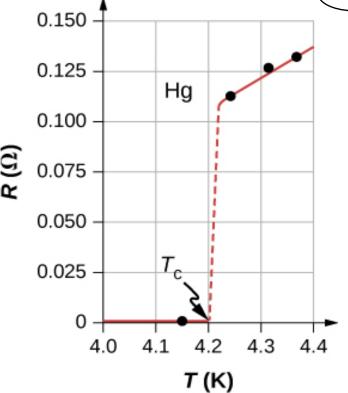
- A brief history of Nb₃Sn
- From bronze to internal Sn wires: the multifilamentary wires age
- J_c and B_{c2} are essential, yet not sufficient for applications
- FCC-hh and the new challenges for Nb₃Sn

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Discovery of superconductivity

Discovery of superconductivity in 19

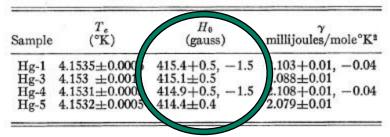


Superconductivity will make possible high-field magnets soon! OK, maybe not so soon!

Heike Kamerlingh Onnes



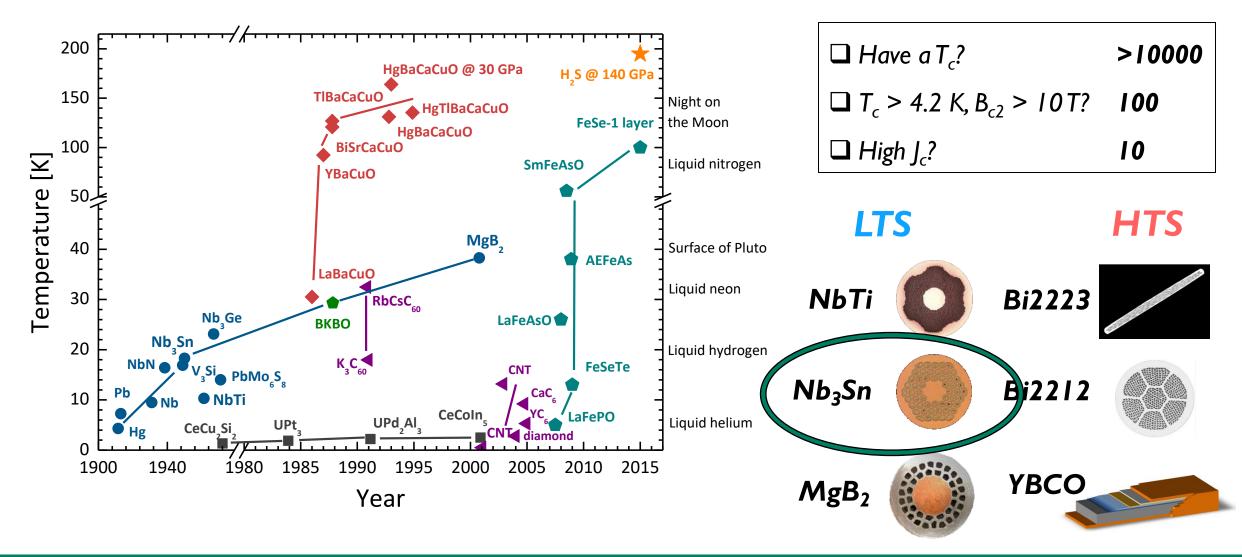
Table I. Critical constants for superconducting mercury.



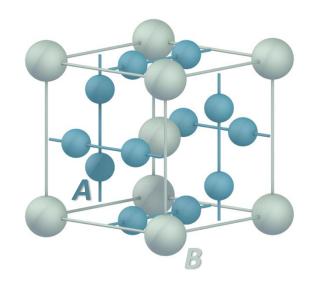
M. Wilson *IEEETAS* (2012) 22 3800212

D. K. Finnemore et al. Physical Review (1960) 1181

Superconductors are not created equal



A15 compounds



The name originates from Strukturbericht notation with chemical formula A₃B

More than 50 superconductors, 10 of them with $T_c \ge 15 \text{ K}$

 V_3 Ga, Nb_3 Sn and Nb_3 Al can be produced as practical conductors

 V_3 Si is the earliest example, $T_c = 17.1$ K (Hardy, 1954)

PHYSICAL REVIEW

VOLUME 93, NUMBER 5

MARCH 1, 1954

The Superconductivity of Some Transition Metal Compounds*

George F. Hardy† and John K. Hulm Institute for the Study of Metals, University of Chicago, Chicago, Illinois (Received November 23, 1953)

About eighty transition metal compounds comprising borides, carbides, nitrides, oxides, silicides, and germanides of metals of Groups 4A, 5A, and 6A were tested for superconductivity down to 1.20°K, using a magnetic method. Among the specimens were most of the known compounds of the above type not examined magnetically for superconducting behavior by previous workers, and in all cases the structures were checked by a real diffraction analysis. The following eleven new superconductors were discovered, in parentheses: W₂B (3.10°), Nb₂C (9.18°), Ta₂C (3.26°), O. 9. Mo₃Ce (1.43°), a. ThSi; (3.44°).

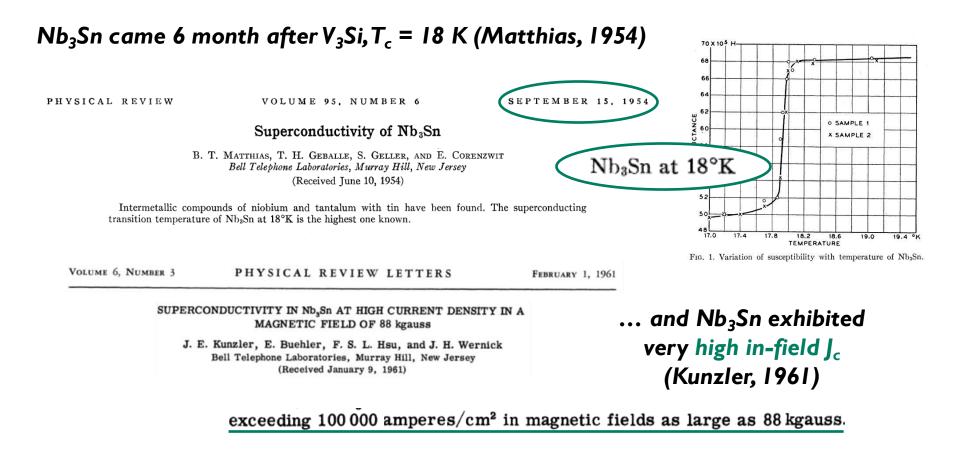
V₃Si (17.1°)

2), Mo₃Ge (1.43°), α-ThSi₂ (3.16°), β-ThSi₂ (2.41°) perconducting germanides, V₂Ge and Mo₂Ge, which coic β-tungsten structure. The transition temperature try superconducting compound.

Nb_3 Ge held the record for the highest T_c (23.2 K) until 1986

	Ti	Zr	V	Nb	Та	Cr	Мо
Al			11.8 K	18.8 K			0.6 K
Ga			16.8 K	20.3 K			0.8 K
In			13.9 K	9.2 K			
TI				9.0 K			
Si			17.1 K	19.0 K			1.7 K
Ge			11.2 K	23.2 K	8.0 K	1.2 K	1.8 K
Sn	5.8 K	0.9 K	7.0 K	18.0 K	8.4 K		
Pb		0.8 K		8.0 K	17.0 K		
As			0.2 K				
Sb	5.8 K		0.8 K	2.2 K			
Bi		3.4 K		4.5 K			
Тс							15.0 K
Re							15.0 K
Ru						3.4 K	10.6 K
Os			5.7 K	1.1 K		4.7 K	12.7 K
Rh			1.0 K	2.6 K	10.0 K	0.3 K	
lr	5.4 K		1.7 K	3.2 K	6.6 K	0.8 K	9.6 K
Pd			0.08 K				
Pt	0.5 K		3.7 K	10.9 K	0.4 K		8.8 K
Au		0.9 K	3.2 K	11.5 K	16.0 K		

Nb₃Sn: an old material still to be unraveled



B.T. Matthias et al. Physical Review (1954) 95.6 1435

J. E. Kunzel et al. Physical Review Letters (1961) 6.3

Adapted from R. Flükiger et al. Cryogenics (2008) 48 293

Nb₃Sn: the first high field Nb₃Sn magnet



Magnet, fabricated and tested by: L. Martin, C. Bruch, M. Benz and C. Rosner (left to right) Worked only ONCE!

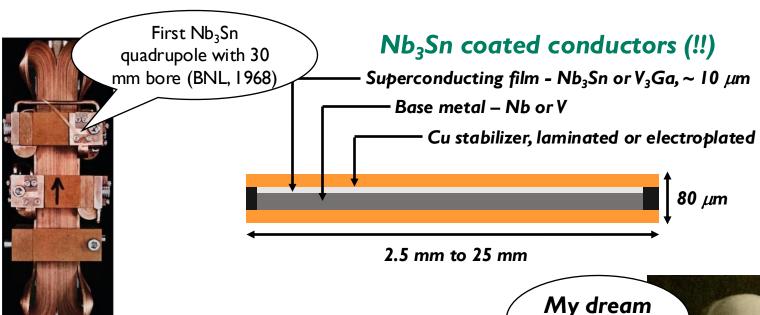
B.T. Matthias et al. Physical Review (1954) 95.6 1435

L. Rossi and A.V. Zlobin Nb₃Sn Accelerator Magnets (2019): 53

M. G. Benz IEEE Trans. Mag. (1966) 2 760

H. Rosner et al. IEEE-CSC ESAS Eur. Supercond. News Forum. No. 9. (2012)

E. Barzi and A.V. Zlobin Nb₃Sn Wires and Cables for High-Field Accelerator Magnets (2019)



Liquid Diffusion Process:

Nb ribbon coated with Sn and heat treated at 900-1200°C for several hours

Developed at General Electric and used in 15T-class R&W magnets

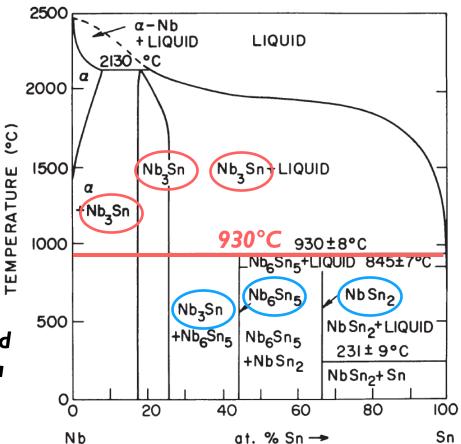


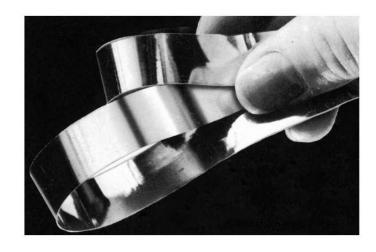


comes true

Nb₃Sn: a complicated family

Nb₃Sn is the only stable compound above 930 °C



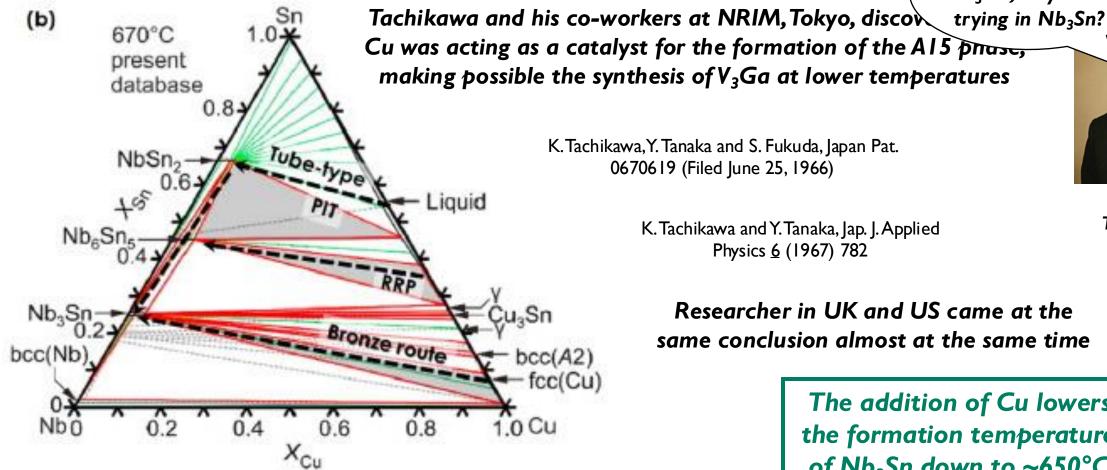


Below 930 °C also Nb₆Sn₅ and NbSn₂ are stable, and have a better kinetics of formation

The reaction of Nb and Sn to form Nb_3Sn needs to be performed at $T > 930^{\circ}C$

D. P. Charlesworth et al., J. of Mat. Sci. (1970) 5 580

Not only Nb and Sn



If it works for V_3 Ga, why not

K. Tachikawa, Y. Tanaka and S. Fukuda, Japan Pat. 0670619 (Filed June 25, 1966)

> K. Tachikawa and Y. Tanaka, Jap. J. Applied Physics <u>6</u> (1967) 782

Prof. Kyoji **TACHIKAWA**

Researcher in UK and US came at the same conclusion almost at the same time

> The addition of Cu lowers the formation temperature of Nb₃Sn down to ~650°C

Not only Nb and Sn

Ta and Ti enhance

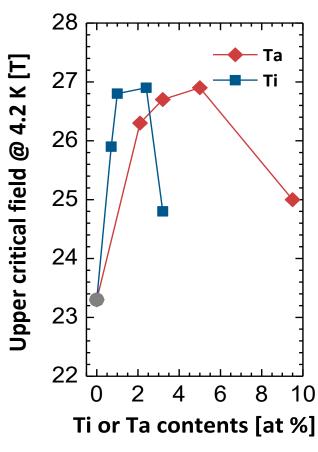
B_{c2} and now are

used in all

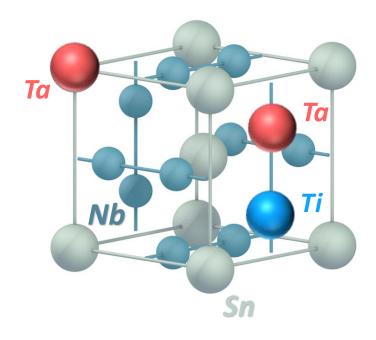
industrial wires

$$B_{c2} \propto \gamma \rho_n T_c$$

 B_{c2} is proportional to defects



Adapted from M. Suenaga et al., JAP <u>59</u> (1986) 840



From EXAFS investigations

- Ta substitutes both Nb and Sn
- Ti substitutes Nb

S. M. Heald et al. Sci. Rep. (2018) 8 4798

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Why multifilamentary wires are important

Nb₃Sn tape, Cu plated

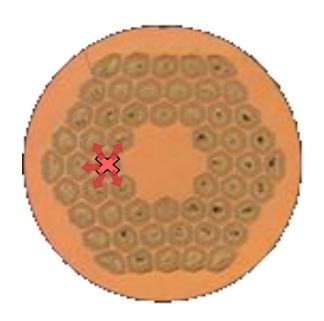
Sn deficiency, defects, etc.

Cracks

What happens?

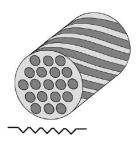
Quench!

The sudden and undesirable transition from a superconducting state to a normal state. When a quench occurs, the stored energy in the superconductor is rapidly converted into heat



In a multifilamentary wire, in case of cracks or defects, J_c can be shared by adjacent filaments!

Filaments can be twisted to avoid current coupling in case the matrix resistivity is low



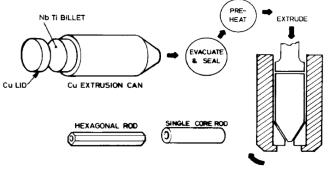
- Minimize AC losses
- Reduce coil magnetisation
- Improve electromechanical stability

Multifilamentary wire are more "stable"!

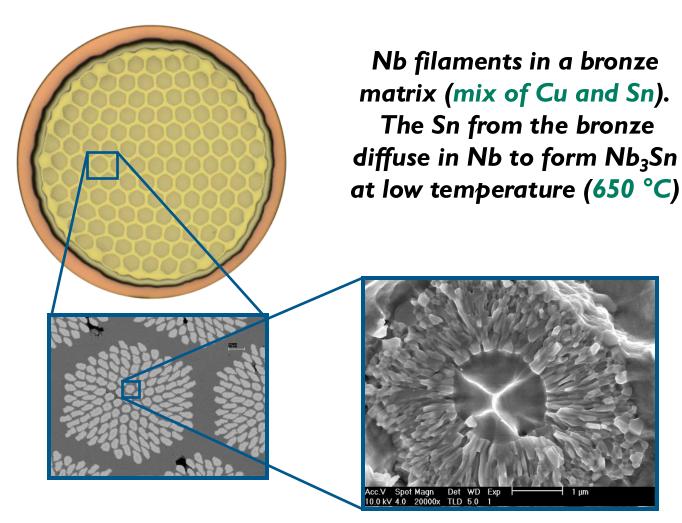
The bronze route and multifilamentary wires

Bronze ingots for Nb₃Sn wires (Courtesy of Osaka Alloying Works Co)

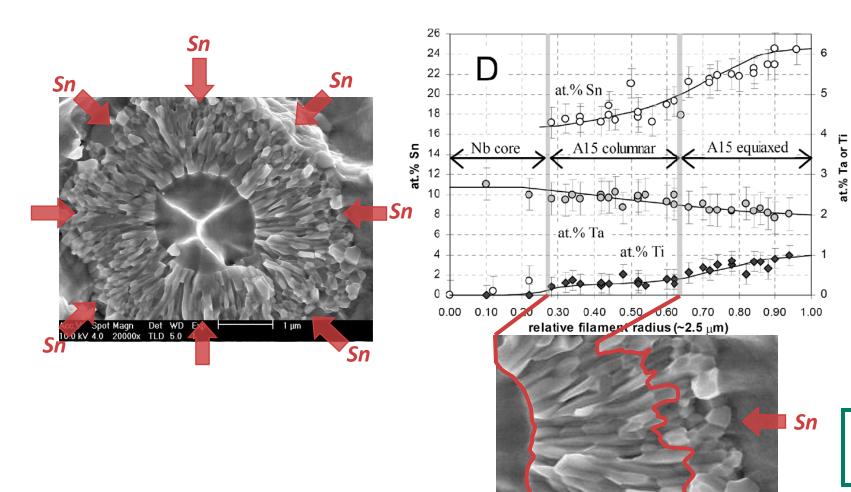




K.Tachikawa and P. Lee, Nb₃Sn and related A15s, Chapter 11 E.W. Howlett, Great Britain Pat. 52, 623/69 (Filed Oct. 27, 1969) A. R. Kaufman and J. J. Pickett, Bull. Am. Phys. Soc. (1970) 15 833



Different grains, different properties



Sn content changes drastically along the Nb₃Sn layer

Equiaxed grains: 21-25 at.% Sn

Columnar grains: 18-21 at.%

Also grain size is different

Equiaxed grain size ~150 nm

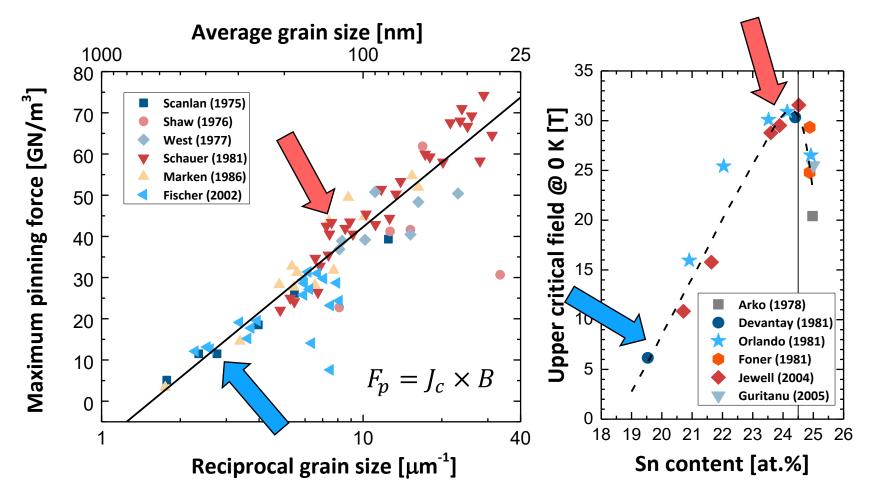
Columnar grain size up to 400 nm

Effect due to low Sn content in bronze

Does these differences affect superconducting properties?

V. Abächerli et al. IEEE TAS (2005) 15 3482

Different grains, different properties

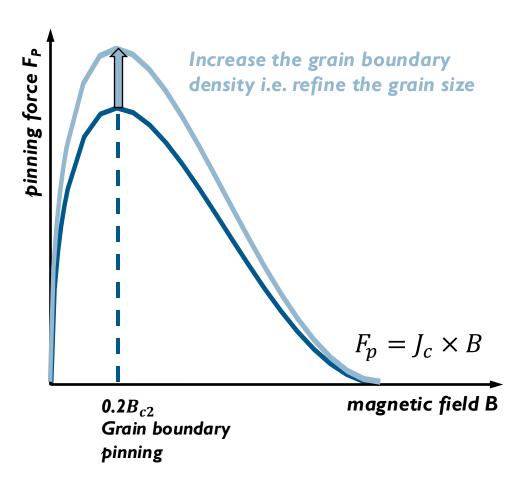


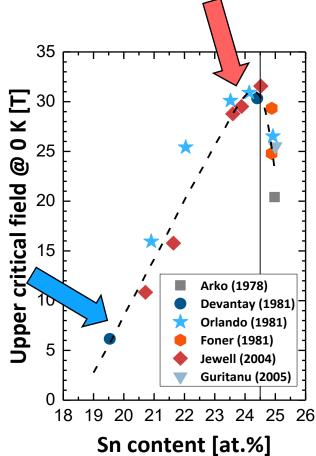
Columnar grains (18-21 at.% Sn, 400 nm) have lower B_{c2} and contribute less to J_c then equiaxed grains (21-25 at.% Sn, 150 nm)

Why J_c increase at finer grain size?

Adapted from A. Godeke, SUST (2006)19 R68

Different grains, different properties





Columnar grains (18-21 at.% Sn, 400 nm) have lower B_{c2} and contribute less to J_c then equiaxed grains (21-25 at.% Sn, 150 nm)

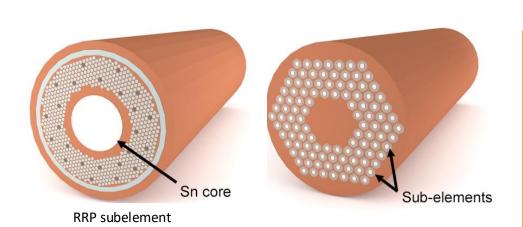
Why J_c increase at finer grain size?

The flux line lattice is pinned by pinning centers (e.g., grain boundaries). The more grain boundaries, the higher the F_p

How to have more Sn and good deformability?

Adapted from A. Godeke, SUST (2006) 19 R68

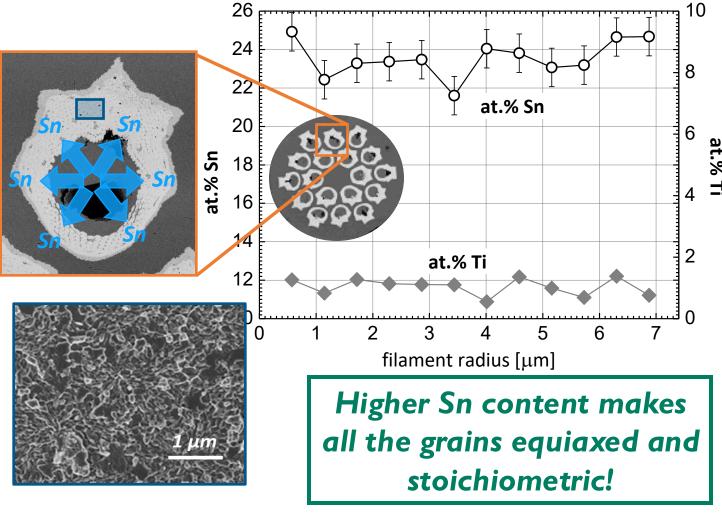
More Sn for the internal Sn



Bronze is substituted by a Sn rod in a Cu matrix

Sn and Cu are deformed easily Sn content can be maximized

Internal Sn includes different type of wires. The most notable are the Rod Restack Process (RRP) wires

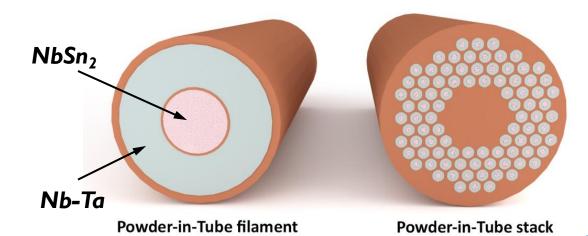


Pictures from C. Sanabria, PhD Thesis (2017), FSU

Another wire fabrication method:

Powder In Tube (PIT)

Sn content decreases linearly along the filament radius

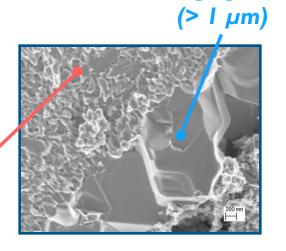


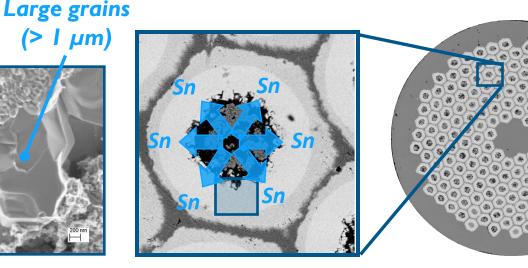
28 26 24 22 20 18 0.0 0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5 6.0 Distance from A15:Core Interface, μm

A Nb tube is filled with NbSn₂ powders, which is used as Sn source in the subelement

Fine grains (~ 200 nm)'
~ 23 at.% Sn

Pictures from C. Sanabria,
PhD Thesis (2017), FSU





Performances in industrial wires



Bronze Process

Bronze is the Sn source, limited by the solubility of Sn in Cu

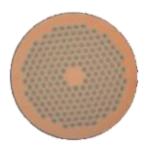
V. Abächerli et al. IEEE TASC (2007) 17 2564



Internal Sn / RRP

A metallic Sn rod is inserted in the subelement core

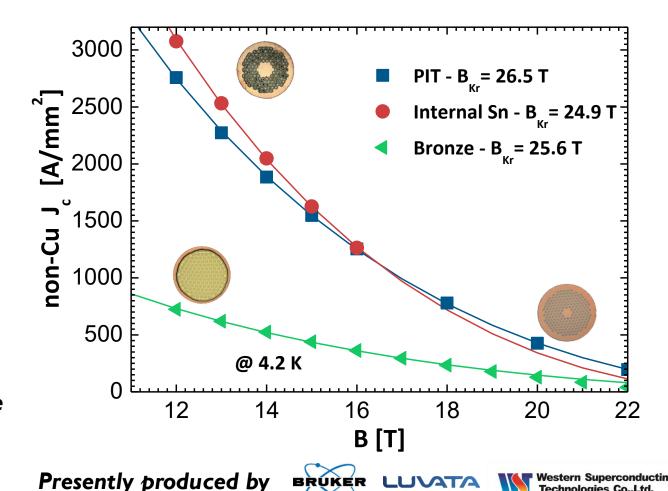
J. Parrell et al. AIP Conf. Proc. (2004) 711 369



Powder-In-Tube (PIT) method

Each subelement is a Nb-alloy tube filled with NbSn₂ and Sn powders

T. Boutboul et al. IEEE TASC (2009) 19 2564





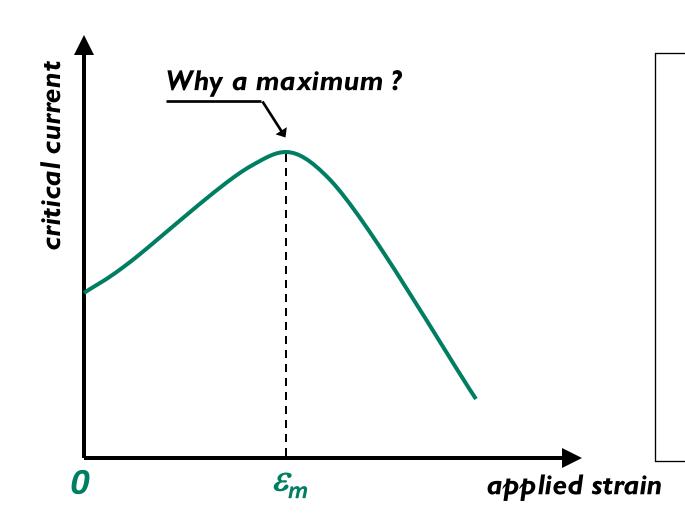


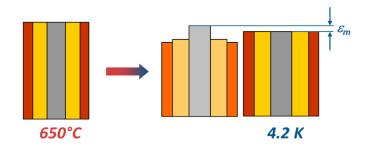


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How axial strain modify Nb₃Sn wires J_c

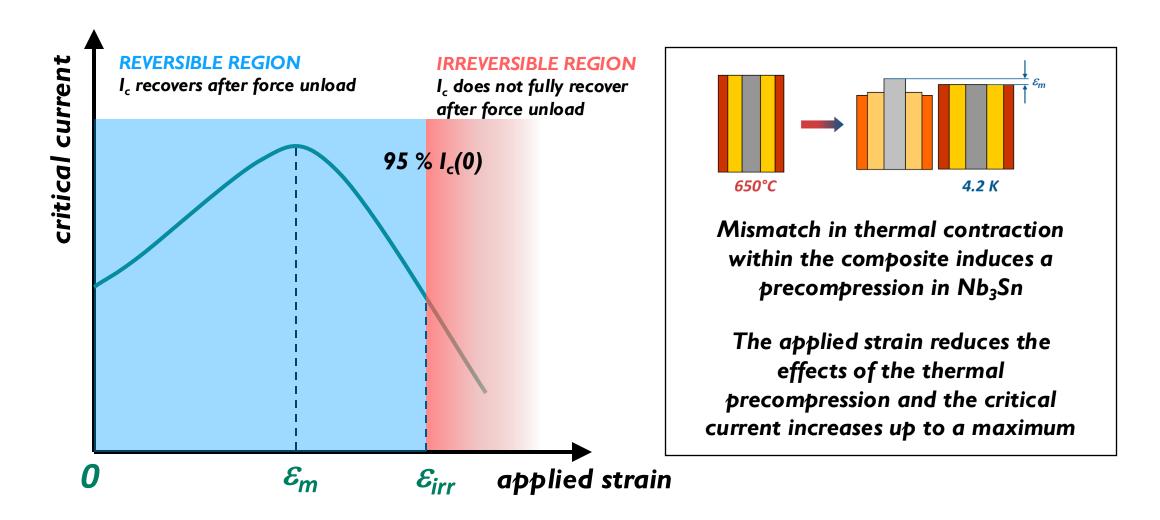




Mismatch in thermal contraction within the composite induces a precompression in Nb₃Sn

The applied strain reduces the effects of the thermal precompression and the critical current increases up to a maximum

How axial strain modify Nb₃Sn wires J_c

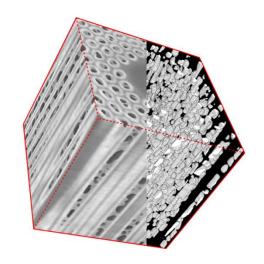


Irreversible degradation phenomena

Two mechanisms govern the irreversible degradation of the critical current

I - Formation of cracks in the Nb₃Sn filaments due to the stress concentration

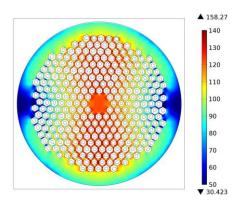
2 - Plastic deformation of the matrix and residual stress on the Nb₃Sn filaments.



I – Axial tension and the role of cracks at the voids

Analysis of the phenomenon

in two load geometries

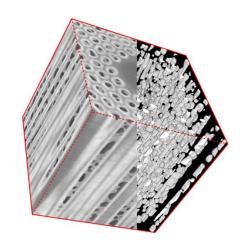


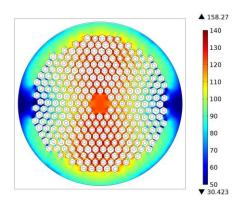
2 – Transverse compression, plastic deformation of the matrix and residual stress on Nb₃Sn

Analysis of the phenomenon in two load geometries

I - Axial tension and the role of cracks at the voids

2 – Transverse compression, plastic deformation of the matrix and residual stress on Nb₃Sn

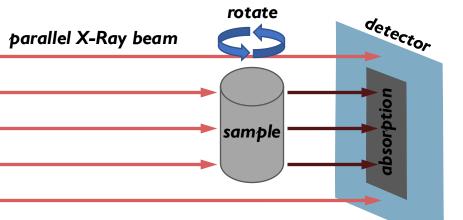




Voids detection in Nb₃Sn wires



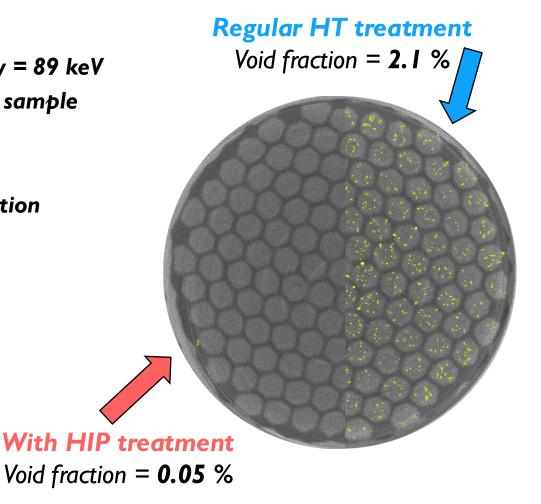
X-ray microtomography reconstruction @ ESRF Grenoble



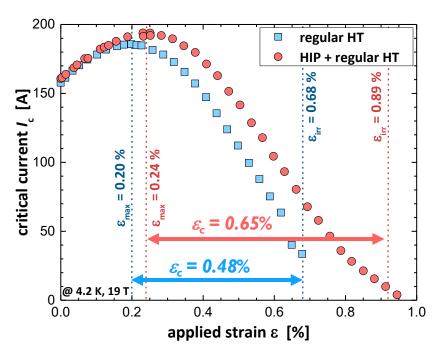
- X-ray photon energy = 89 keV
- 360° rotation of the sample
- 30'000 projections
- 2560 x 2160 pixels
- 0.57 μm/pixel resolution



Non-destructive 3D volume reconstruction with separation of internal features to study voids distribution



Irreversible limit measurement

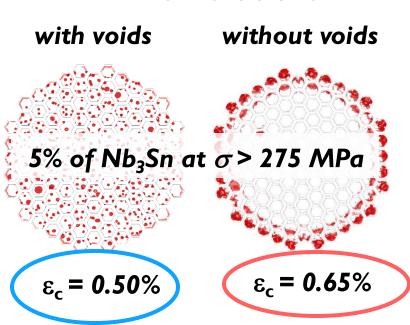


Regular HT:
$$600^{\circ}$$
C/100h + 670° /150h
$$\varepsilon_{c} = \varepsilon_{irr} - \varepsilon_{max} = 0.48 \%$$

HIP 550°CHh/200MPa + Regular HT
$$\varepsilon_{c} = \varepsilon_{irr} - \varepsilon_{max} = 0.65 \%$$

With HIP treatment ε_c increases by +0.17 %

FEM simulations



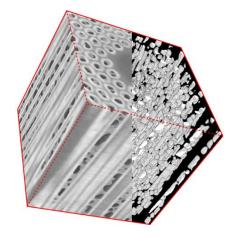
C. Barth et al. Sci. Rep. (2018) 8 6589

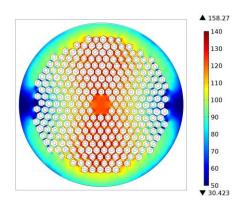
Changes in the voids correlate quantitatively with the changes in the electromechanical limits

Analysis of the phenomenon in two load geometries

I – Axial tension and the role of cracks at the voids

2 – Transverse compression, plastic deformation of the matrix and residual stress on Nb₃Sn





Design options for the FCC-hh dipoles

h2020 EuroCirCol WP5, started in 2015

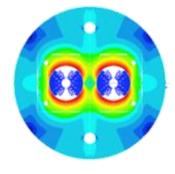


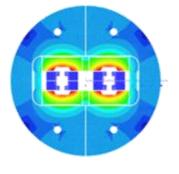


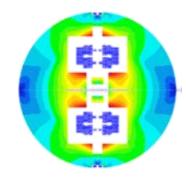


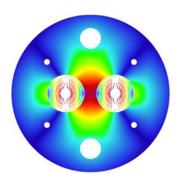












Cosine Theta Coil

Block Coil

Common Coil

Canted Cosine Theta (CCT)

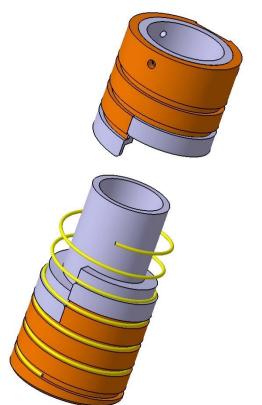
All designs for the 16 T dipoles share a peak in transverse stress at operation of 150-200 MPa



Nb₃Sn Rutherford cable for HL-LHC, 40 strands

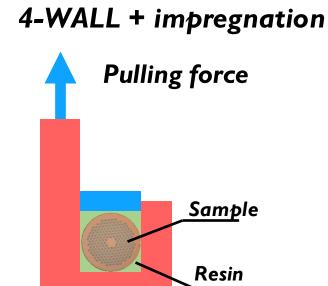
Are the Nb₃Sn wires in the Rutherford cables able to withstand such a high stress level? Which degradation is tolerable?

The WASP concept



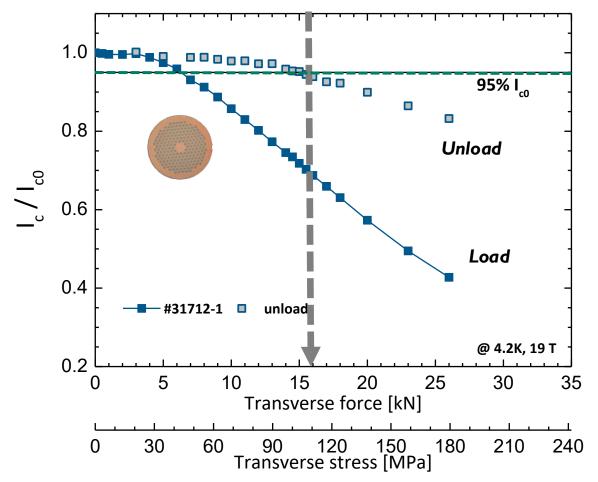


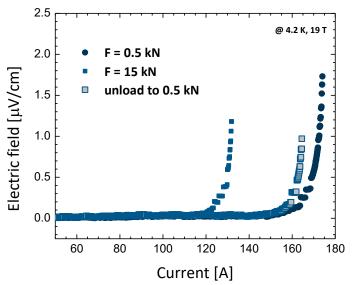
B. Seeber et al. IEEE TASC (2007) 17 2643G. Mondonico et al. SuST (2012) 25 115002





collaboration agreement KI 629/TE (2009-2012)





The irreversible limit is defined at the force level leading to a 95% recovery of the initial I_c after unload

Here

$$F_{irr}(B=19T)=16 \text{ kN}$$

The corresponding irreversible stress limit is

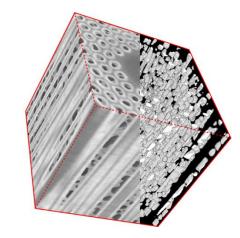
$$\sigma_{irr}(B=19T) = 110 \text{ MPa}$$

where

Two mechanisms govern the irreversible degradation of the critical current

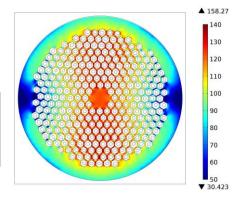
☐ Formation of cracks in the Nb₃Sn filaments due, for instance, to the stress concentration at the voids formed during the reaction heat treatment

Cracks generate a reduction of the current carrying cross section \Rightarrow I_c^{unload}/I_{c0} is independent of the magnetic field

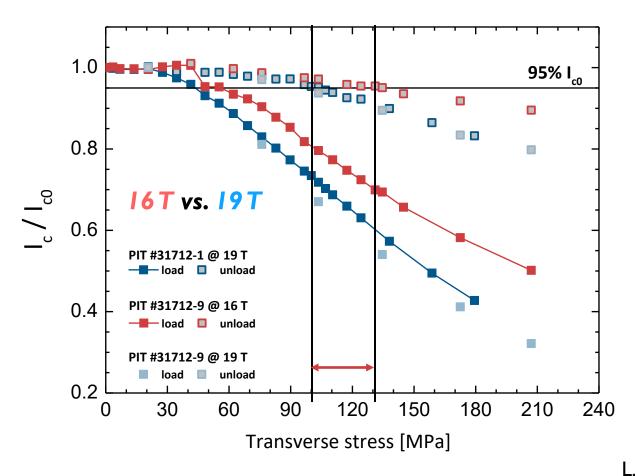


□ Plastic deformation of the matrix and residual stress on the Nb₃Sn filaments.

Residual stress induces a permanent reduction of B_{c2} after unload $\Rightarrow I_c^{unload}/I_{c0}$ depends on of the magnetic field



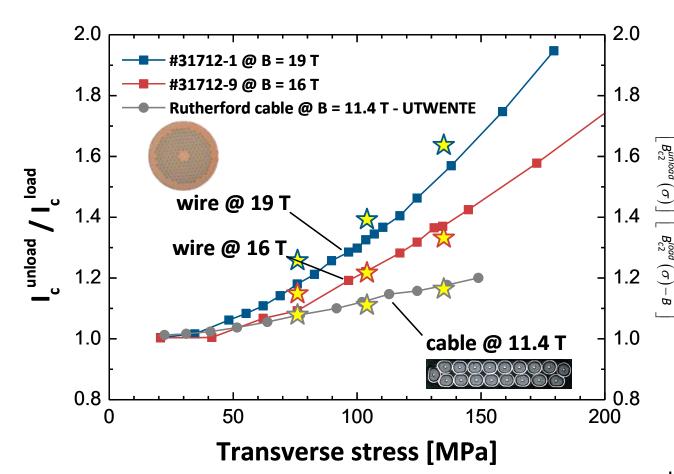
Field dependence of the irreversible stress limit



Clear field dependence!

L. Gamperle et al. Physical Review Research (2020) 2.1 013211.

Field dependence of the irreversible stress limit



Clear field dependence!

A simple model, based only on the effects of residual stress, reproduces the experimental dependences on field and stress

It proves also that the experiments performed on the single wire are consistent with those on cables

$$\frac{I_c^{unload}}{I_c^{load}}(B,\sigma) = \left[\frac{B_{c2}^{load}(\sigma)}{B_{c2}^{unload}(\sigma)}\right]^{\frac{3}{2}} \left[\frac{B_{c2}^{unload}(\sigma) - B}{B_{c2}^{load}(\sigma) - B}\right]^2$$

L. Gamperle et al. Physical Review Research (2020) 2.1 013211.

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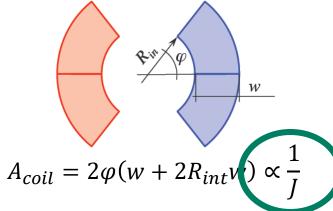
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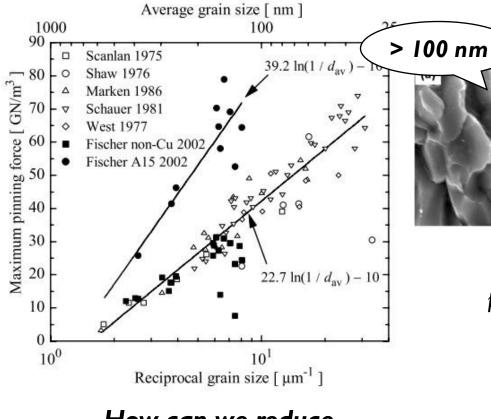
The Future Circular Collider



LHC 27 km, 8.33 T 14 TeV (c.o.m.) 1'300 tons NbTi FCC-hh 91 km, 14 T ~ 85 TeV (c.o.m.) ~10'000 tons Nb₃Sn

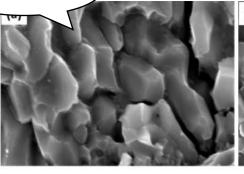
A huge amount of Nb₃Sn!!

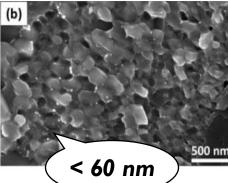




How can we reduce effectively grain size?

Internal oxidation!





Grain refinement induced by precipitation of oxides nanoparticles

X. Xu et al. Appl. Phys. Lett. (2014) 104, 082602

DOI:10.1063/1.4866865.

M. G. Benz, <u>Trans. Met. Soc. AIME</u>, (1968) <u>242: 1067-70</u>.

L. E. Rumaner et al. Metall Mater Trans A (1994) 25, 213–219

DOI: <u>10.1007/BF02646689</u>.

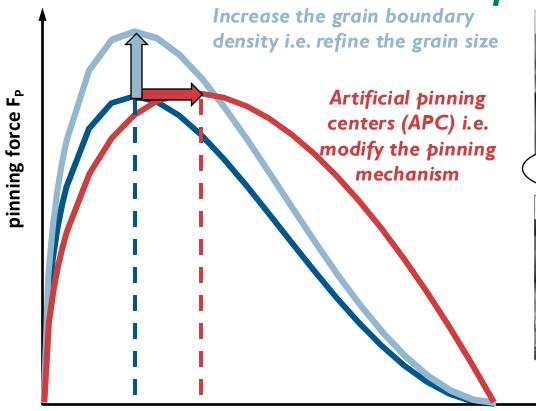
A. Godeke, Supercond. Sci. Technol (2006) 19 R68

DOI: <u>10.1088/0953-2048/19/8/R02</u>

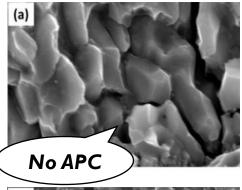
E. Todesco, IEEE Transactions on Applied Superconductivity

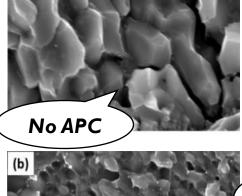
(2025) **DOI:**10.1109/TASC.2025.3558196

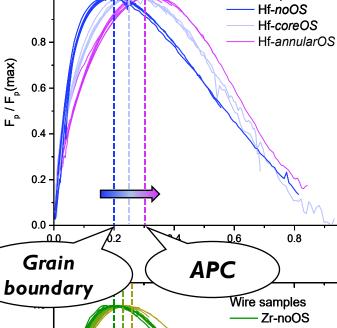
More then just a F_{b} increase



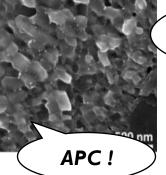


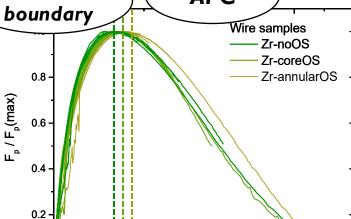






Wire samples



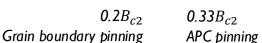


0.4

B/B₂₂ (1 %)

0.2

0.0



$$F_p = J_c \times B$$

Internal Oxidation leads to grain

magnetic field B

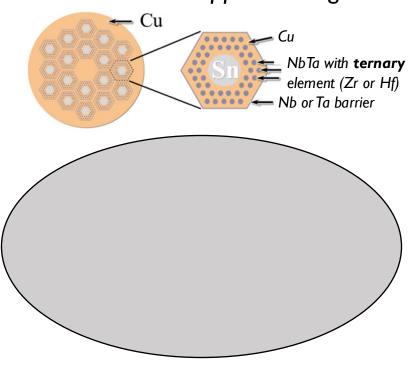
refinement AND produces **Artificial Pinning Centers (APCs)**

8.0

0.6

Internal oxidation process

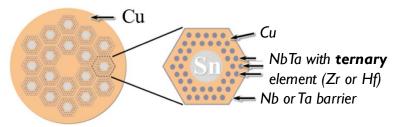
What happens during heat treatment

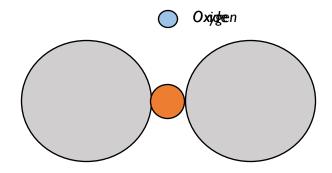


During heat treatment, grains grows and join together when their boundaries touches

Internal oxidation process

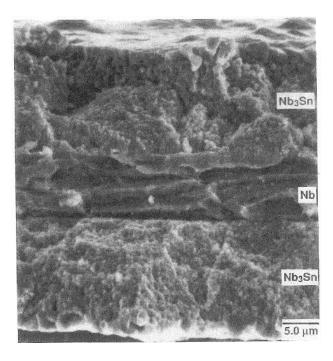
What happens during heat treatment





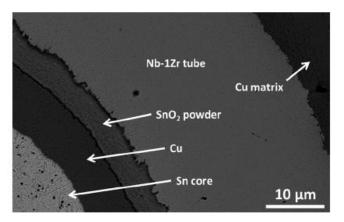
Oxygen reacts with Hf or Zr (higher oxygen affinity)

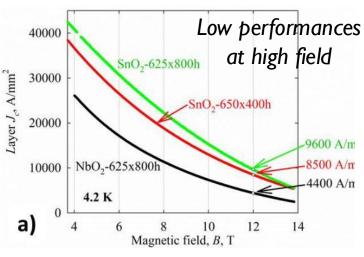
ZrO₂ (or HfO₂)
nanoparticles inhibits grain
boundaries movement



Proposed in 1968 on tape samples, the year before bronze route was developed. Forgotten for a long time as considered impossible to be implemented in wires

The first wire was fabricated in 2014, using a separate oxygen source to allow wire deformation



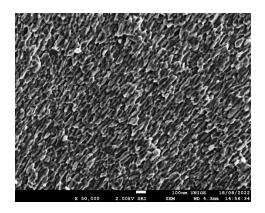


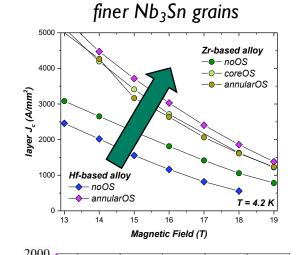
M. G. Benz Transaction of the Metal. Soc. of AIME (1968) 242 X. Xu et al. Applied Physics Letters (2014) 104.8 082602)

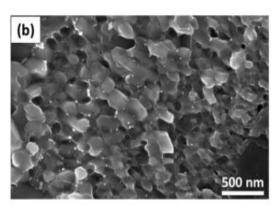
The benefits of internal oxidation

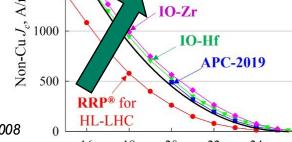
Higher I induced by

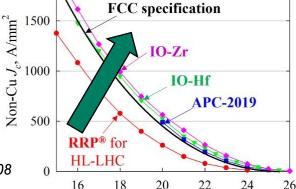
Grain refinement induced by precipitation of oxides nanoparticles





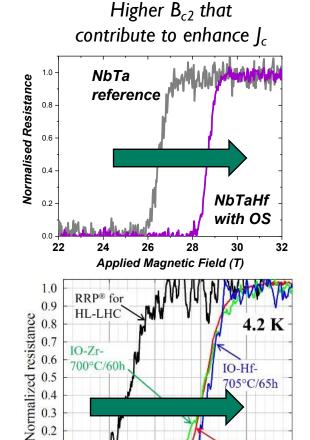






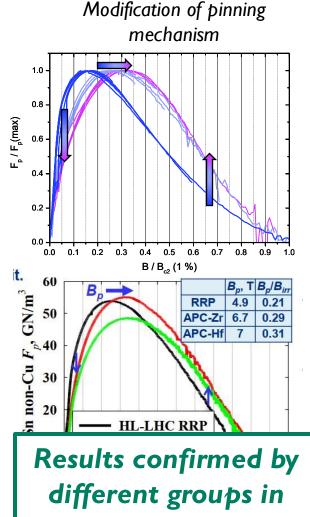
Magnetic field, B, T

4.2 K



Magnetic field, B, T

APC2019-



X. Xu et al. Supercond. Sci. Technol. (2023) 36 085008

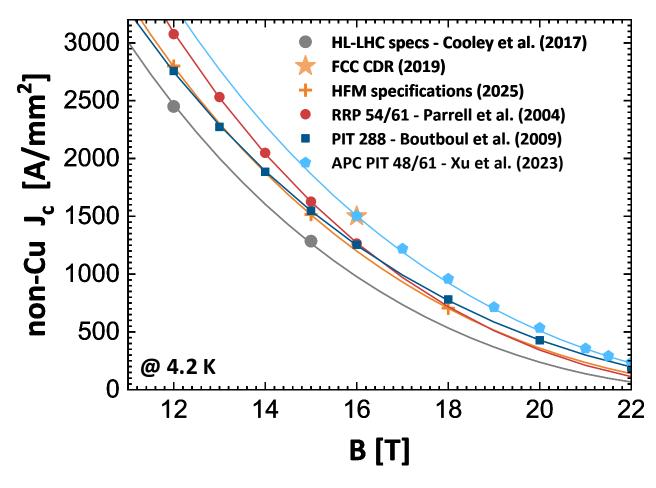
DOI: 10.1088/1361-6668/acdf8c.

X. Xu et al. Supercond. Sci. Technol. (2023) 36 035012

DOI: 10.1088/1361-6668/acb17a

multiple studies

Specifications for FCC-hh



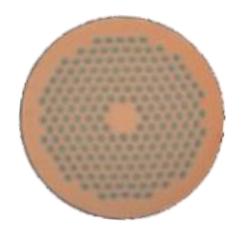
The best non-Cu J_c values for RRP and PIT wires meet the HFM specifications

Prototype PIT wires with internal oxidation (APC) exhibit even higher non-Cu J_c

Improved conductor performance could either reduce the coil size at a given field or enable higher fields

Cooley et al., IEEE TASC <u>27</u> (2017) 6000505 Abada et al., Eur. Phys. J. Special Topics <u>228</u> (2019) 755–1107 Todesco, HFM Forum (2025), URL: <u>indico.cern.ch/event/1501797/</u> Parrell et al., AIP Conf. Proc. <u>711</u> (2004) 369 Boutboul et al., IEEE TASC <u>19</u> (2009) 2564 Xu et al., Supercond. Sci. Tech. <u>36</u> (2023) 035012

Manufacturing process: RRP vs PIT

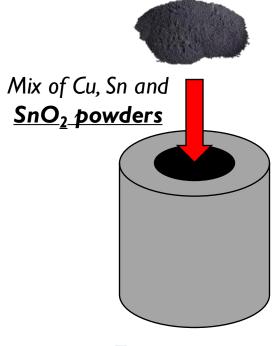


Nb₃Sn multifilamentary wire are not created equal!

The nature of the wire subunit (Subelement) matters!

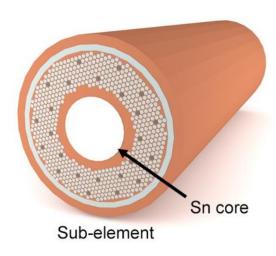
Remember, internal oxidation requires the addition of oxide powders!

PIT wires



Easier route to introduce powders

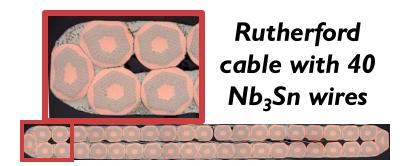
RRP wires



Powders not present in wire layout

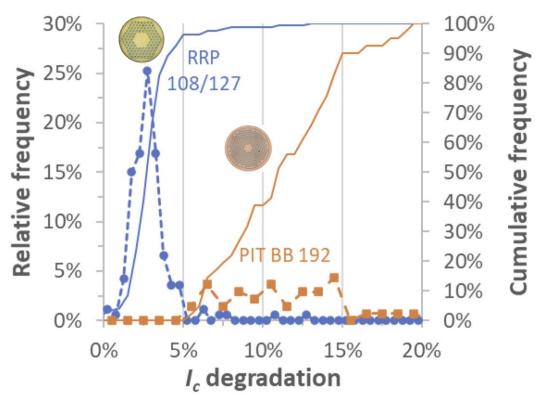
Pictures from C. Sanabria, PhD Thesis (2017), FSU

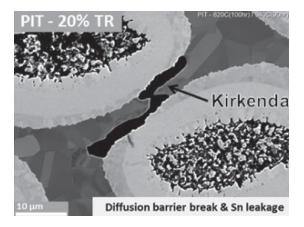
Manufacturing process: RRP vs PIT





Section of a Nb₃Sn dipole magnet





Distorted filaments produce Sn leak. Reaction is not complete, leading to the lower I_c and Cu contamination

Lower cabling degradation in Internal Sn RRP wires compared to PIT wires

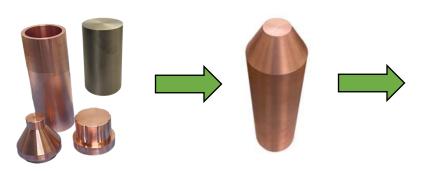
S. Hopkins et al. IEEE Trans. Appl. Supercond. <u>34</u> (2024) 6001308 **DOI:** 10.1109/TASC.2024.3375274

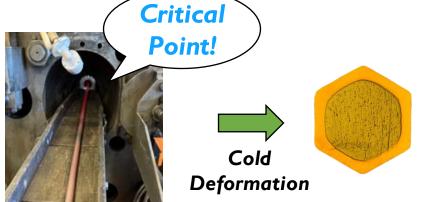
M. Brown et al. Supercond. Sci. Technol. <u>29</u> (2016) 084008 **DOI:** <u>10.1088/0953-2048/29/8/084008</u>

C. Segal et al. Supercond. Sci. Technol. <u>29</u> (2016) 085003 **DOI:** 10.1088/0953-2048/29/8/085003

How it's made: high-J_c RRP Nb₃Sn wire



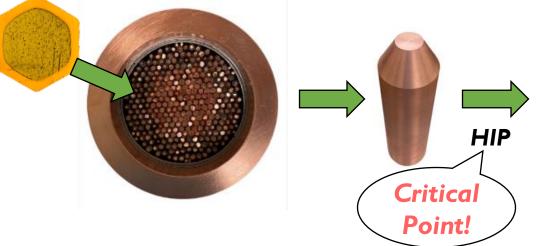


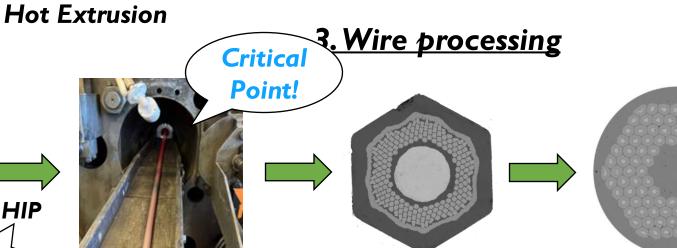


Hot Extrusion

RRP wire production involves hot extrusion, hot isostatic pressing and cold deformation





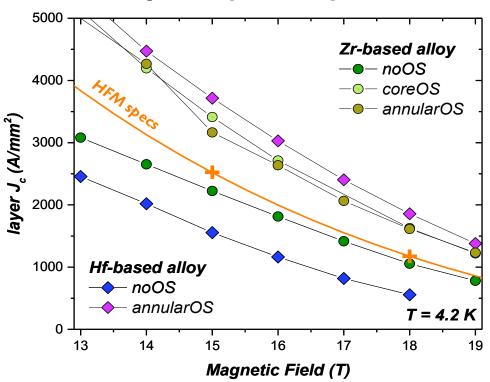


Sn rod insertion and cold deformation

Subelement restack and cold deformation to final diameter

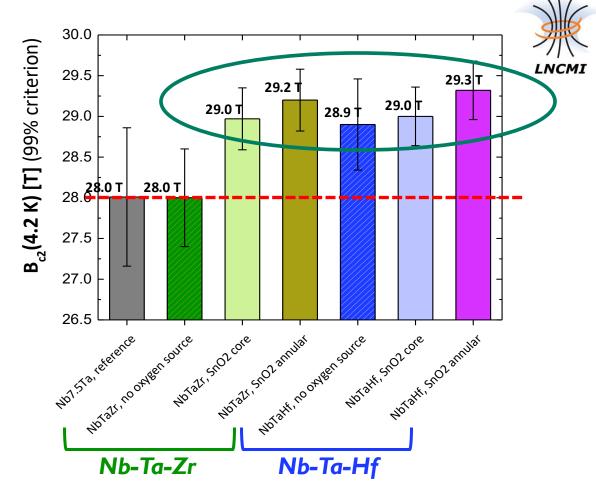
Internal oxidation in simplified RRP wires

Transport I_c and B_{c2} measurements



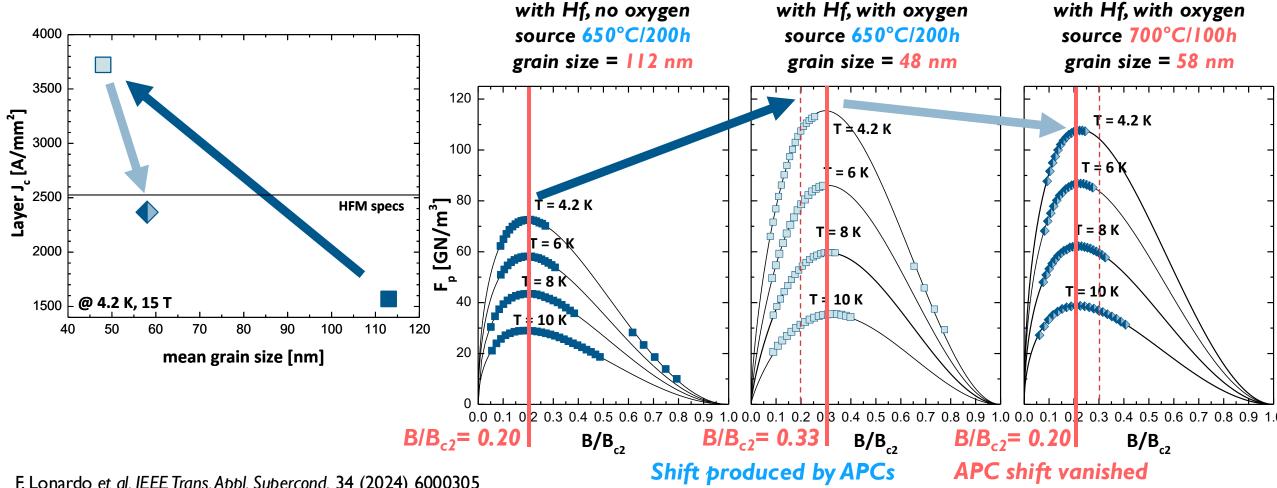
Layer J_c determined from transport measurements

G. Bovone et al. Supercond. Sci. Tech. <u>36</u> (2023) 095018 DOI: <u>10.1088/1361-6668/aced25</u>



R(B) tests performed up to 33 T at LNCMI-Grenoble show record high B_{c2} values achieved both with Hf and Zr

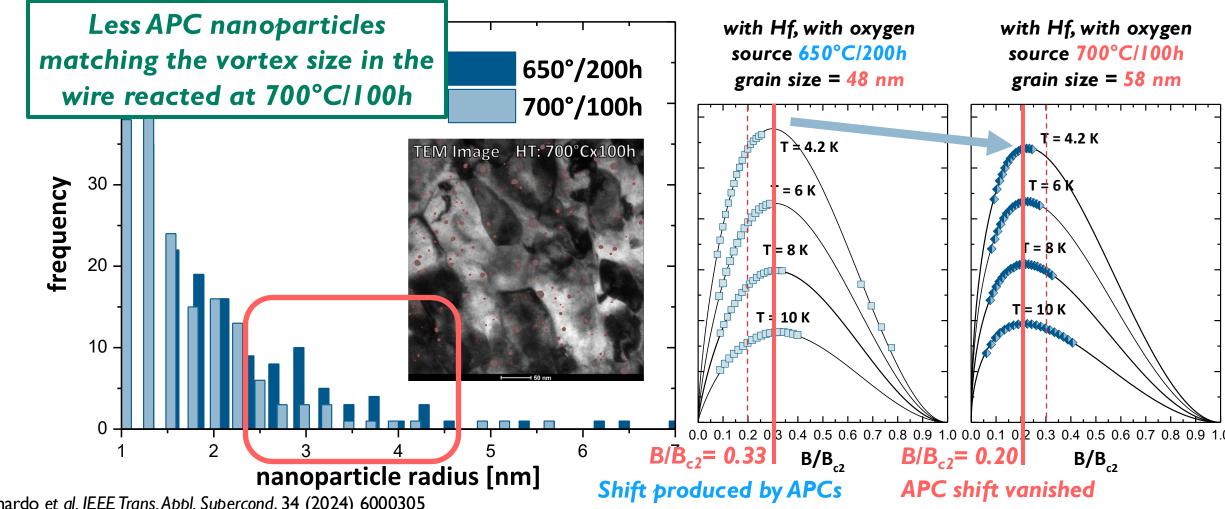
The size of APC nanoparticles matters



F. Lonardo et al. IEEE Trans. Appl. Supercond. 34 (2024) 6000305

DOI: 10.1109/TASC.2024.3355353

The size of APC nanoparticles matters



F. Lonardo et al. IEEE Trans. Appl. Supercond. 34 (2024) 6000305

DOI: 10.1109/TASC.2024.3355353

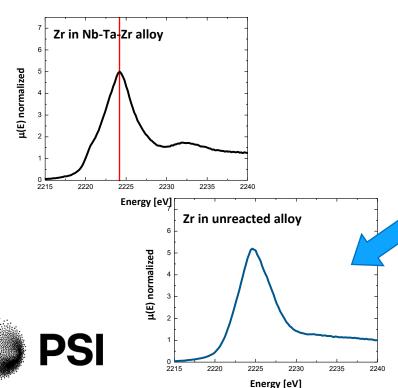
APC nanoparticles forms along with Nb₃Sn

Spatial distribution of the nanoparticles by X-ray Absorption Spectroscopy

during the heat treatment

REFERENCE #1

XAS spectrum of Zr in Nb-Ta-Zr alloy



G. Bovone et al. IEEE Trans. Appl. Supercond. <u>34</u> (2024) 6000205 DOI: 10.1109/TASC.2024.3354232

Reacted Monofilamentary Wire REFERENCE #2 XAS spectrum of Zr in ZrO₂ ZrO_sreference Oxygen diffusion Energy [eV] Zr in Nb_sSn ZrO₂-like **spectrum** found only in Nb₃Sn! Zr spectrum in unreacted alloy very similar to reference #1 despite the oxygen diffusion Energy [eV]

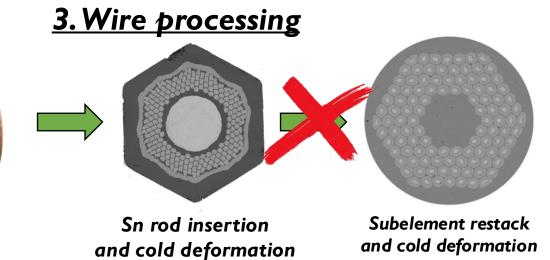
Internal oxidation in extruded subelement

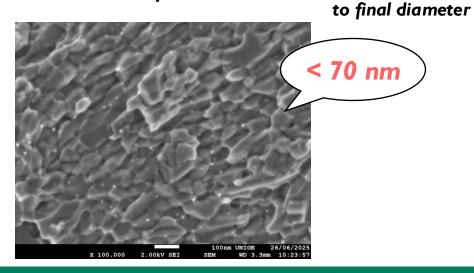


20 mm

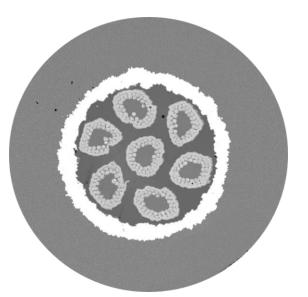
°C. Each filament contains OS at the periphery of Nb

The oxygen source promoted grain refinement of Nb₃Sn





Internal oxidation in a simplified RRP

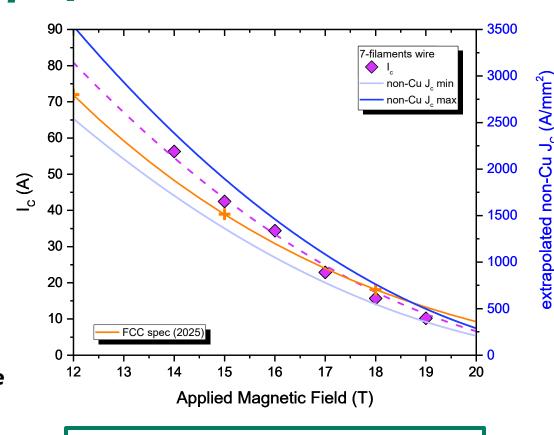


Simplified RRP wire (7/7) made from cold deformed subelements (filaments and subelements are cold deformed)

Wire Properties	Value	Ideal
Reaction %	53.8 %	100 %
Nb/non Cu Area %	50.0 %	50 – 60 %
Nb/Sn at	3.1	3.1 – 3.6
Sn/Sn+Cu wt %	45.1 %	50 – 60 %

Excess of Cu and low Sn content causes the thin reaction layer.

Wide spread results on J_c due to very variable cross-section, low reaction layer and broken subelements



Internal oxidation successfully implemented in a (simplified)
Nb₃Sn RRP multifilamentary wire

To conclude

- Despite its "age" Nb₃Sn is still an important material
- FCC-hh reignite the fundamental research on Nb₃Sn
- The internal oxidation is the best way to meet FCC-hh specification for the wire
- Internal oxidation promote J_c enhancement due to grain refinement, enhancement of B_{c2} and modified pinning mechanism (APC pinning)
- For applications, I_c is not all, as the wire need to be "robust"

Thank you for your attention