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Evolution of nano-particles doping in Nb_3Sn wires

[1]Mattia Ortino

[2]S. Pfeiffer, [1]T. Baumgartner, [3]M. Sumption, [4]X. Xu, [5]X. Peng, [2]J. Bernardi, [1]M. Eisterer

[1]Atominstitut, TU Wien (Vienna, AT)
[2]USTEM, TU Wien (Vienna, AT)
[3]The Ohio State University (Columbus, USA)
[4]Fermilab (Batavia, USA)
[5]Hyper Tech Research Inc. (Columbus, USA)

EASITrain – European Advanced Superconductivity Innovation and Training. This Marie Sklodowska-Curie Action (MSCA) Innovative Training Networks (ITN) has received funding from the European Union's H2020 Framework Programme under Grant Agreement no. 764879

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Outline

- Introduction
  - Nb_3Sn for FCC-hh
  - Internal oxidation method
  - Pros and cons
- Experimental results
  - B_{c2}
  - J_c
  - Microstructure
  - Pinning
  - Local properties: radial inhomogeneities
- Conclusions

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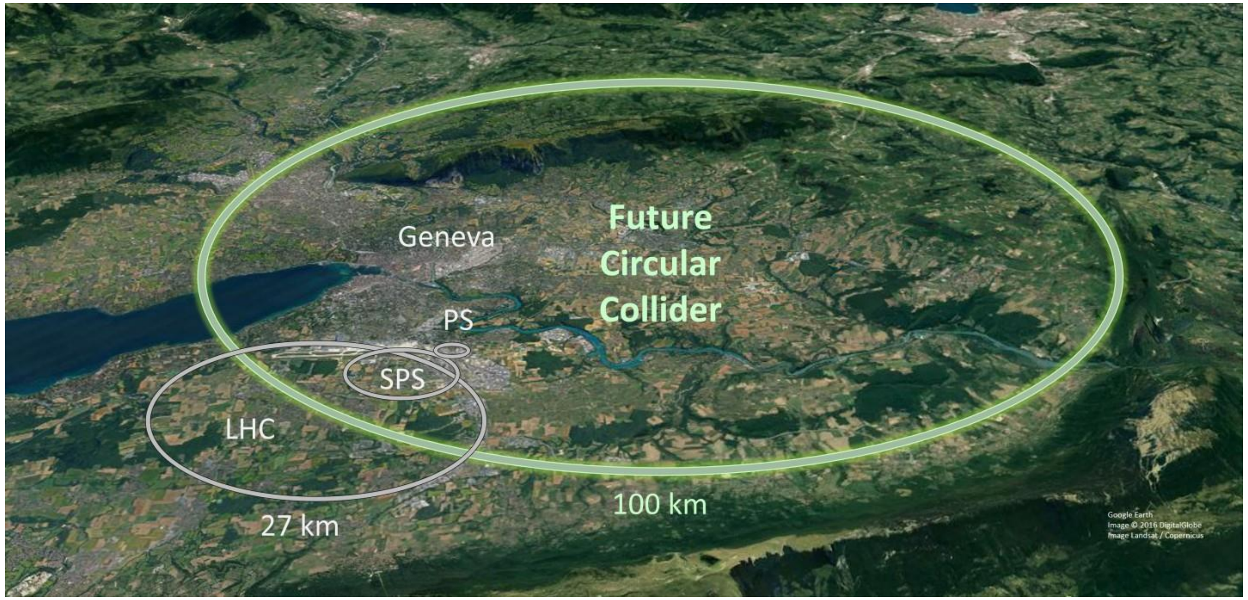
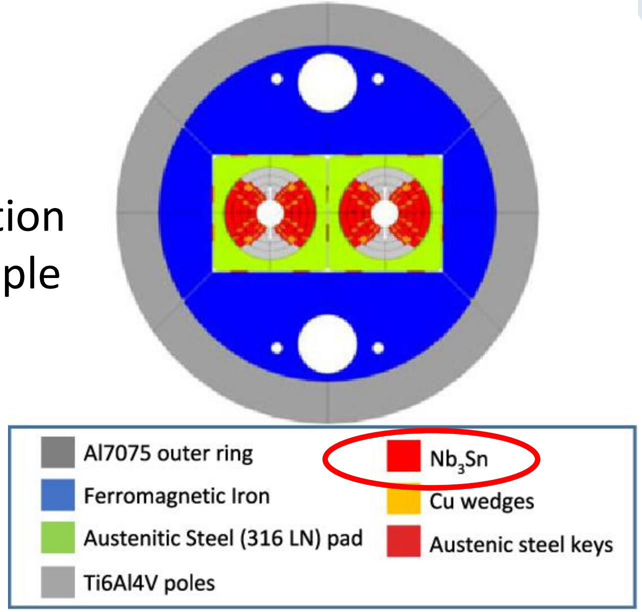
Introduction: Nb_3Sn for FCC-hh

The h(adron)-h(adron) Future Circular Collider (FCC)- CERN, Geneva (CH)



Cosθ – configuration
16T dipole example



Courtesy of M.Boscolo, INFN (IT)

Nb_3Sn: the best conductor candidate

Leading manufacturing technologies → RRP, PIT

	J_c (12 T), RMS [A/mm²]	J_c (15 T), RMS [A/mm²]	B_{c2}(4.3 K), RMS [T]	J_c (16 T), RMS [A/mm²]	J_c (18 T), RMS [A/mm²]	Minimum RRR (15% rolling) -
0.7 mm RRP	**2676**, *68*	**1410**, *58*	**24.5**, *0.39*	**1098**, *55*	**610**, *47*	>100
0.85 mm RRP	**2835**, *44*	**1601**, *33*	**25.9**, *0.19*	**1289**, *30*	**785**, *25*	>100
0.85 mm Bundle Barrier PIT	**2323**, *83*	**1342**, *49*	**26.7**, *0.1*	**1093**, *40*	**688**, *26*	>150

FCC-Goal:

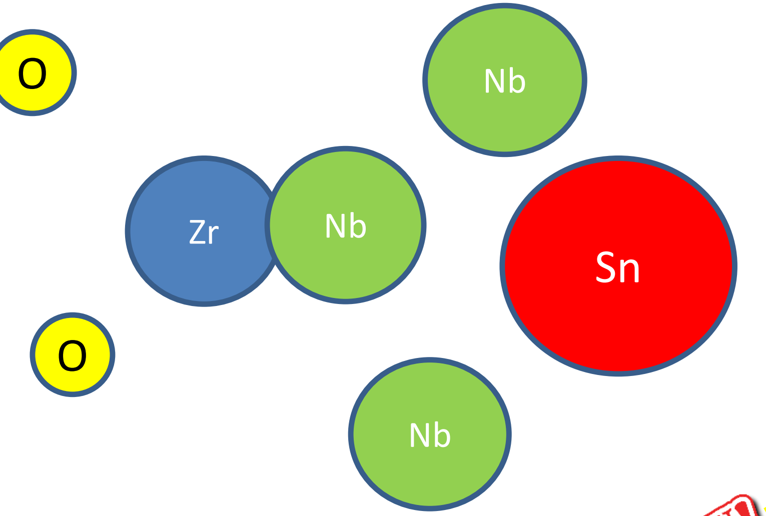
non-Cu J_c=1.5 kA/mm² (16 T, 4.2K)

State-of-the-art Nb_3Sn wires performances insufficient: a final boost is needed

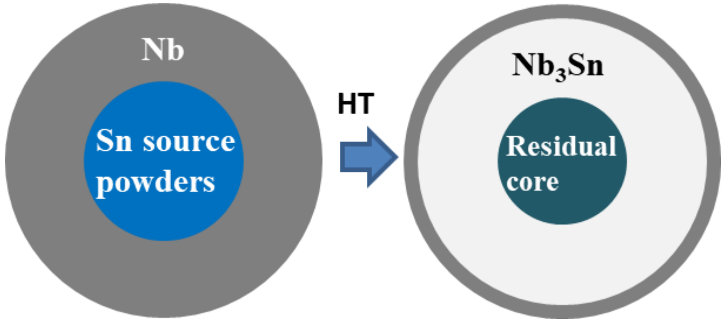
Introduction: [1]Internal oxidation method

[1]X. Xu, M. Sumption, X. Peng, Applied Physics Letters **104**, 082602 (2014)

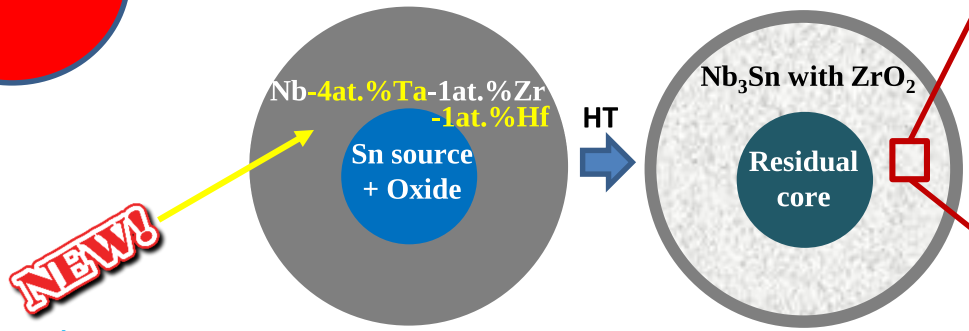
1) O-source → SnO_2 powder (located between the Cu/Sn core and Nb–1Zr)
2) Nb–1wt% Zr alloy → Zr has much stronger affinity to O than Nb:oxidation of the alloy is possible
3) Reaction temperature = 620°-700°

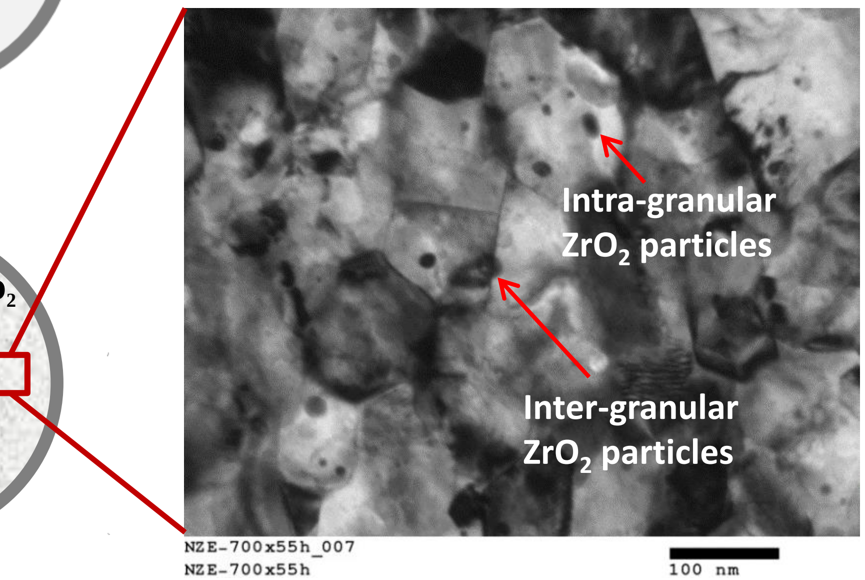


Present-day PIT sub-element:



APC-PIT sub-element:





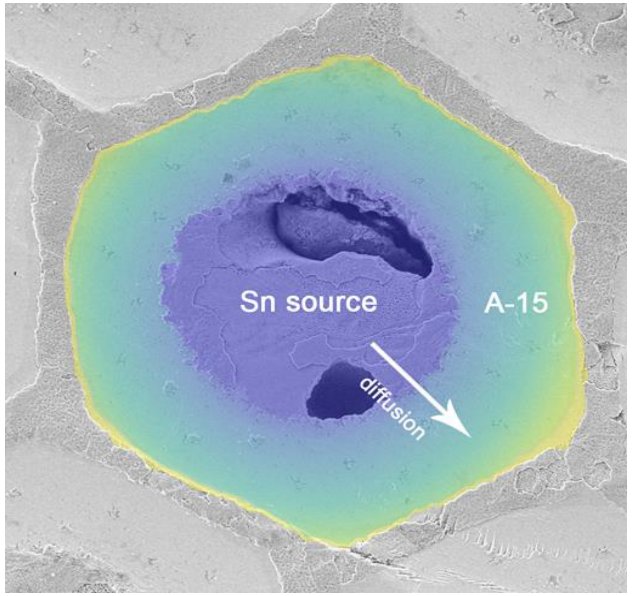
End-2018: From binary to ternary compounds

Introduction: pros and cons

Pros:

- Nanoparticles catalysing A-15 grain size refinement, hereby increasing J_c ($J_c = f(1/dgrain)$);
- ZrO_2 nanoparticles to become as well additional pinning centres (intra-granular);
- Ta addition to raise B_{irr} and B_{c2} of the superconducting phase;

But…Nb_3Sn formed by diffusion reaction: Sn diffuses outwards into a Nb tube (PIT) or -future possibility- in a region containing densely stacked Nb filaments (RRP)



Courtesy of T. Baumgartner, TU Wien

Cons:

- Radial gradient in stoichiometry always present[2], specially in compounds doped with Ta[3]
- Other types of inhomogeneities - sub-element sausaging or barrier breakage- may also occur

Inhomogeneities affect the superconducting performance: B_{c2}, J_c and local properties need to be assessed, relating them to the microstructure

[2]R. Flükiger, C. Senatore, M. Cesaretti et al., Supercond. Sci. Technol. 21 (2008) 054015 (2008)

[3]S. M. Heald , C. Tarantini, et al.,ScieNtific ReporTS | *(2018) 8:4798*

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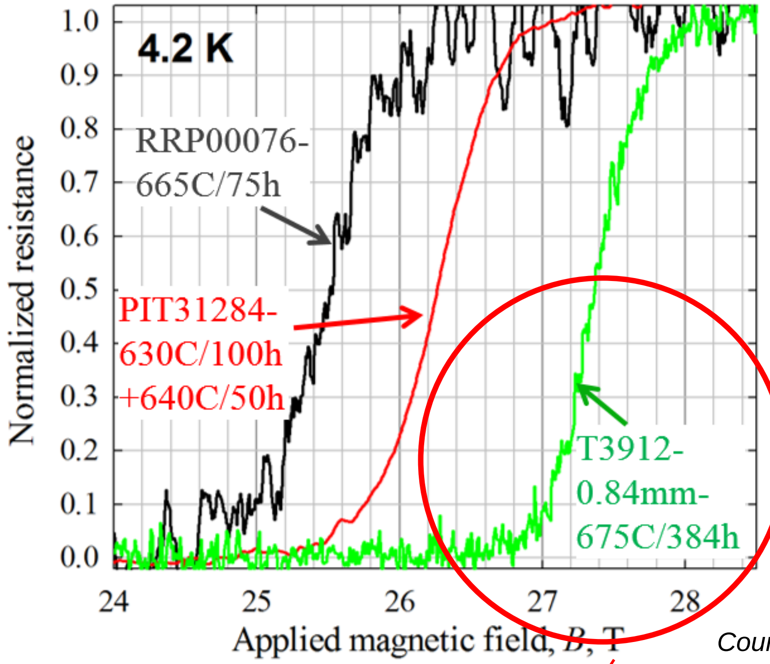
Experimental results: B_{c2}

Binary-samples B_{c2} were measured in a 17 T cryostat via resistive method (values extrapolated to low temperatures)

Still low B_{c2} values (at 4.2 K)

Sample type	B_{c2} [T]
T3607-mono	22.1
T3657-multi	22.6
T3680-mono	23.4
T3682-mono	22.3

Ternary (+ Ta)-samples B_{c2} were measured in a 31T cryostat via resistive method at NHMFL in Jan 2019



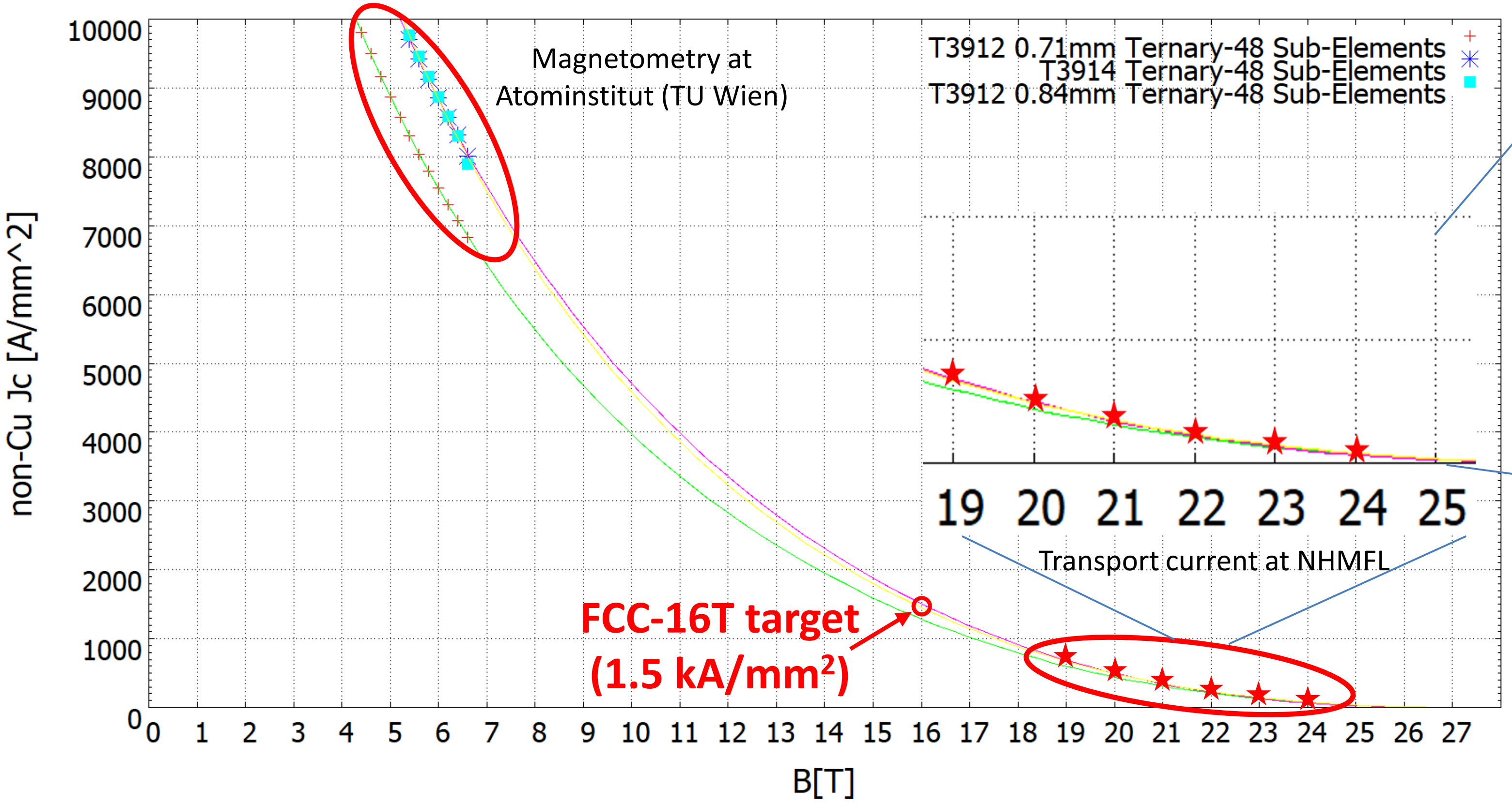
Courtesy of X. Xu, Fermilab

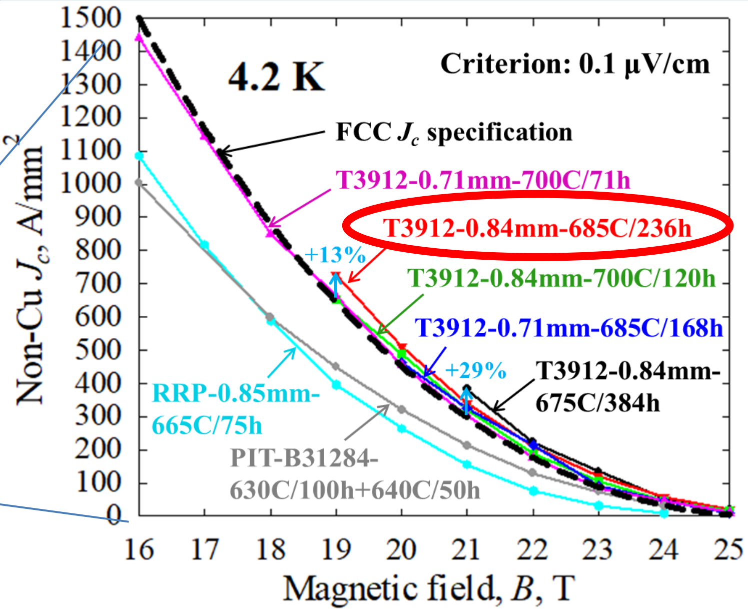
Ta-doping raised B_{c2} of APC-samples (20% to 32%): high field performance of the high-J_c binary samples is expected to improve

Diameter [mm]	Characteristics	B_{c2} [T]
0.71	Nb-4at.%Ta-1at.%Zr tube + SnO2 powders	27.3
0.71	Nb-4at.%Ta-1at.%Hf tube+ SnO2 powders	26.7
0.84	Nb-4at.%Ta-1at.%Zr tube + SnO2 powders	27.8

B_{c2} values used for J_c extrapolation at TU Wien

Experimental results: J_c





Low field data (up to 7 T) measured in a SQUID, then extrapolated to B_{c2} ($Fp(B)$ dependence)

Resistive measurements at NHMFL match the extrapolation: the FCC- target at 16 T is reached!

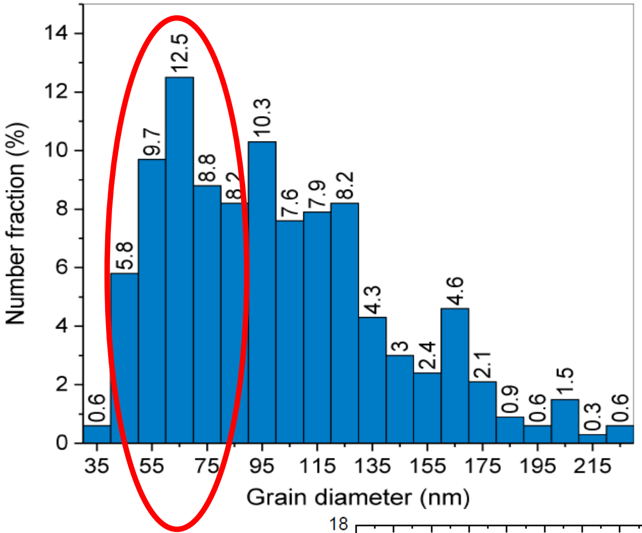
What has been improved?

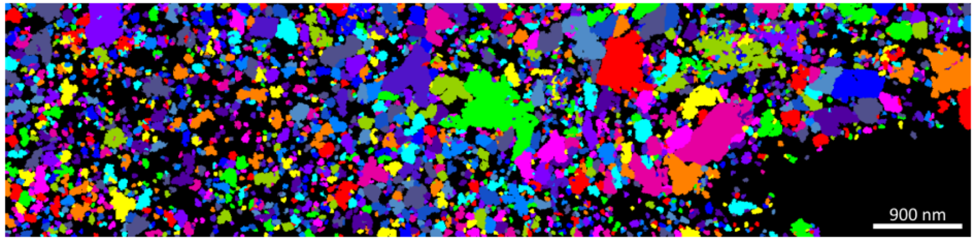
1) Ta gives a different high field behaviour
2) Better Cu/non-Cu ratio: present wires have 1.3 (previous generation had between 2.5 and 3.3)
3) Microstructure? Was the grains size further refined?

Experimental results: microstructure

TEM-Transmission Kikuchi Diffraction (TKD) method

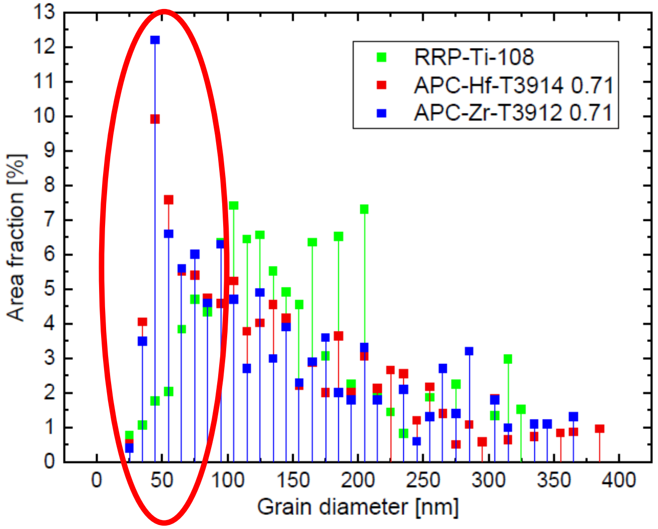
Binaries (TT) best grain size modal value: ~ 60 nm



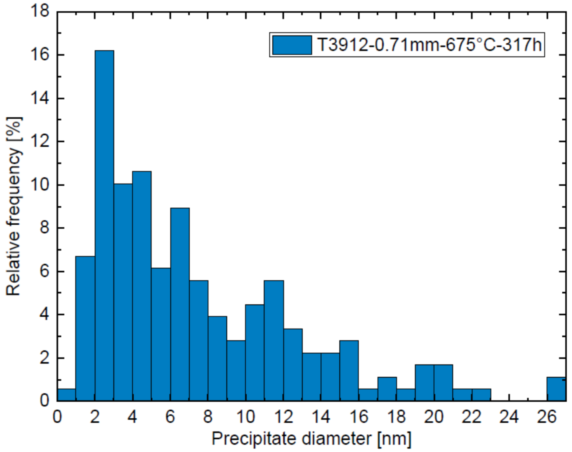


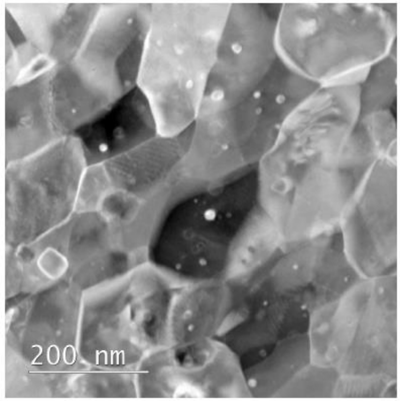
- Grain size refinement not affected by Ta-doping

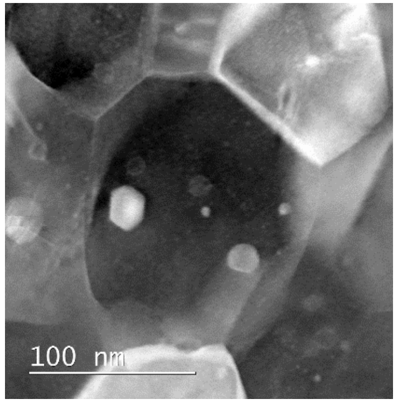
Ternaries (PIT) best grain size modal value: ~ 50 nm

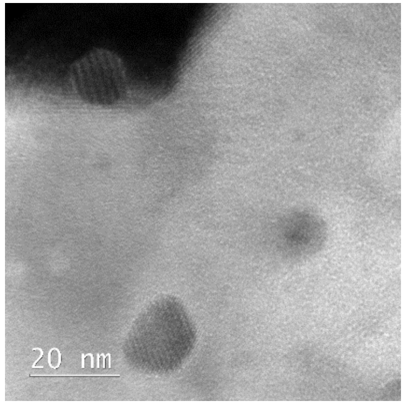


- Likewise, nanoparticles do not show a size change



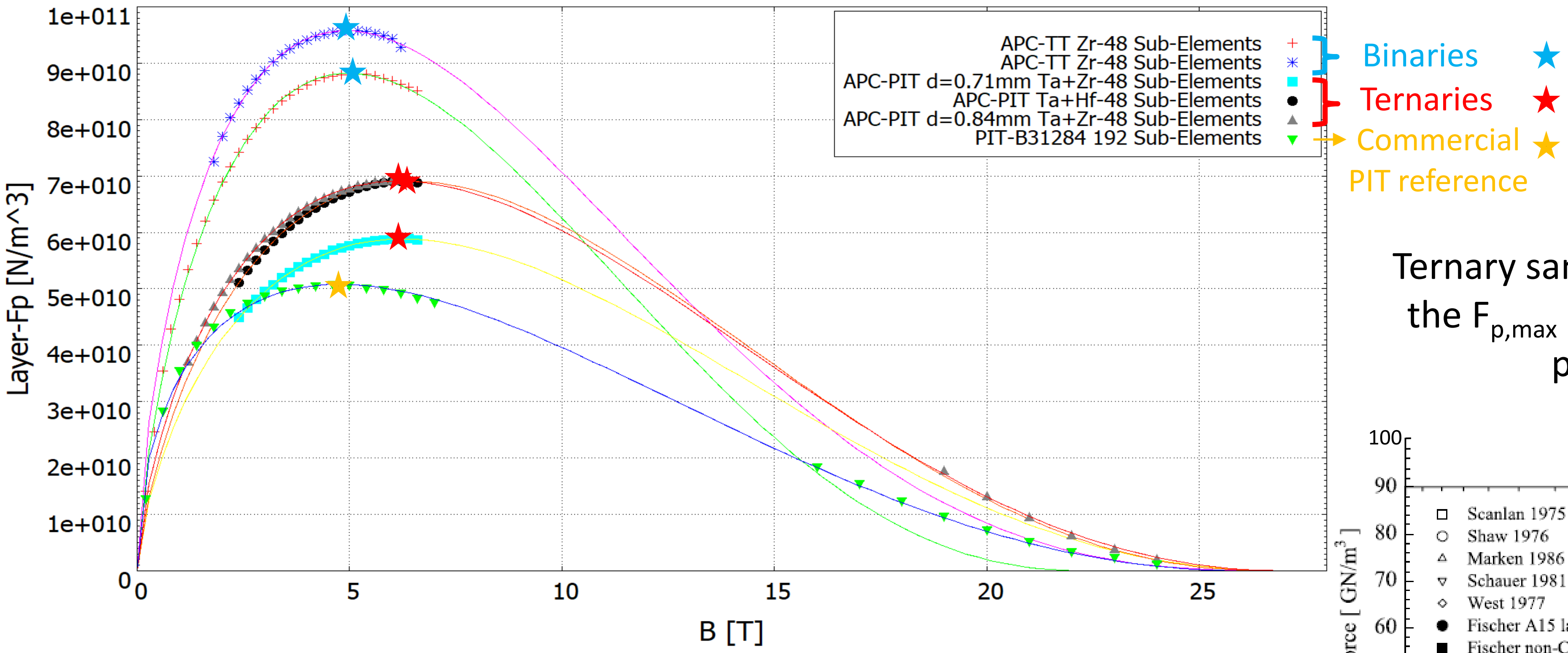




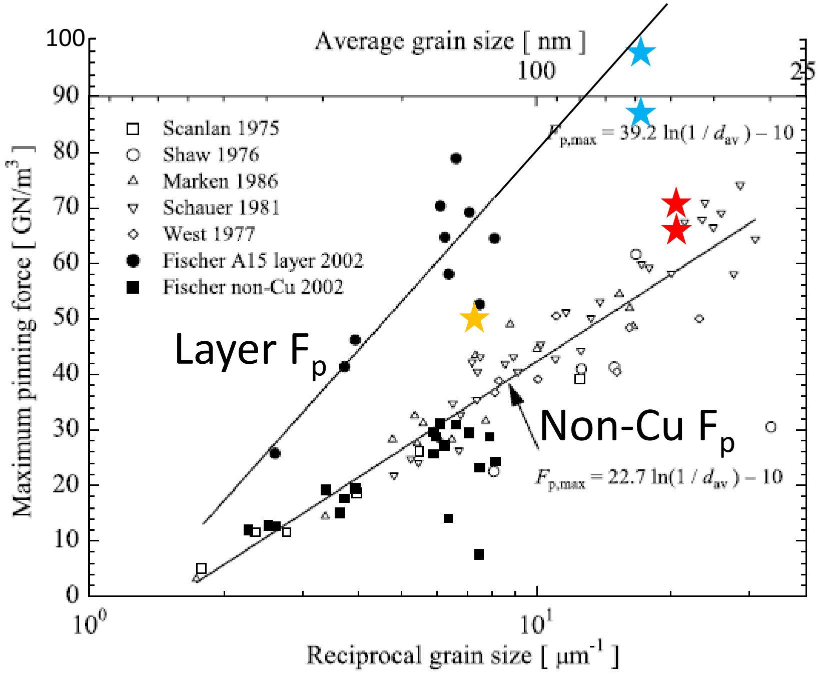


- Still no preferential deposition

Experimental results: Pinning



Ternary samples exhibit a reduction of the $F_{p,max}$ but (as expected) a further peak-shift is visible

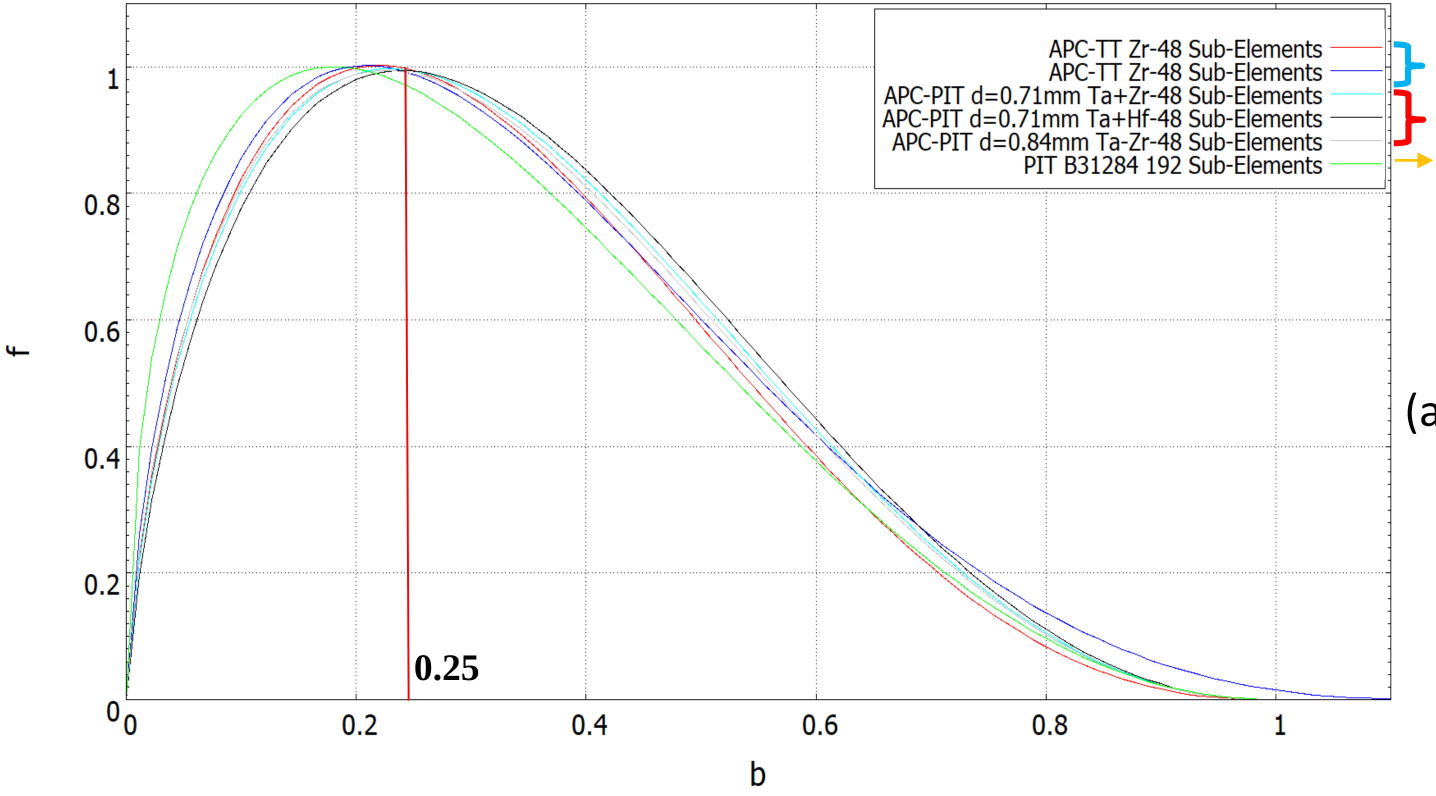


Courtesy of A.Godeke, PhD Thesis (2006)

Single contributions of pure GB-pinning and point-pinning were investigated →

From literature (Bronze-route + PIT), the achieved grain-size refinement would be enough to explain the $F_{p,max}$ values obtained (Commercial-PIT values consistent)

Experimental results: Pinning



Maximum shift in ternaries:
$b(f_{,max})$= 0.25 (Hf-doped samples)

$b(f_{,max})$= 0.22 in binaries

Peak shifts suggest a point pinning contribution (also saw in n-irradiation studies) but its evaluation is difficult

parameters

- $d_{avg} = 4.5\ nm$
- $\rho_{defects} = 25.000\ \mu m^{-3}$
- $l_{avg} \approx 30\ nm$

- Elementary pinning force approach:

$$f_{p,max} \cdot \rho_{defects}$$

$F_{p,max\ (model)}$=51% $F_{p,max\ (exp.)}$

How to weight GB and point pinning contribution still work in progress!

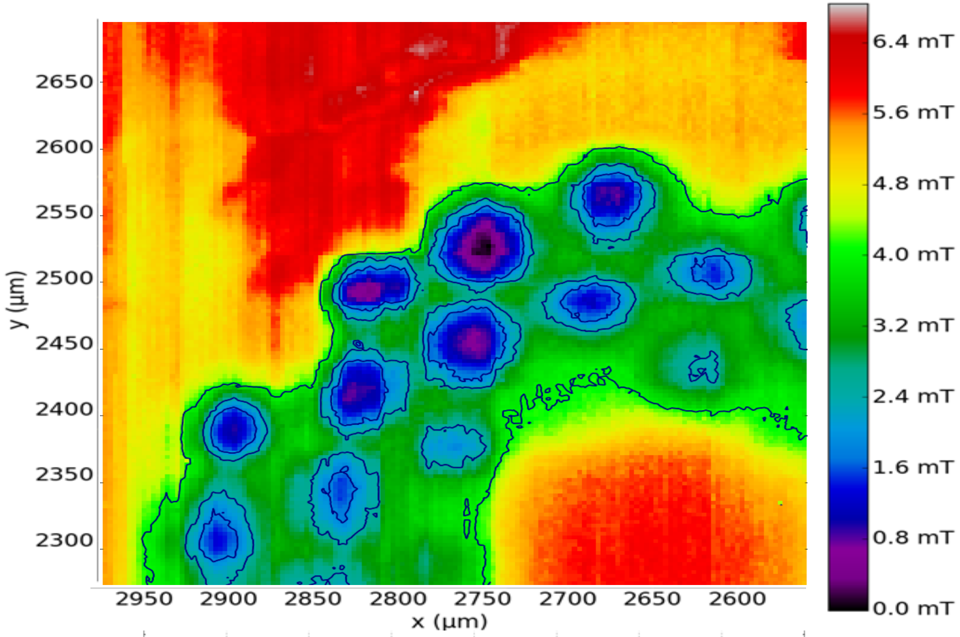
- [4]Dew Hughes approach:

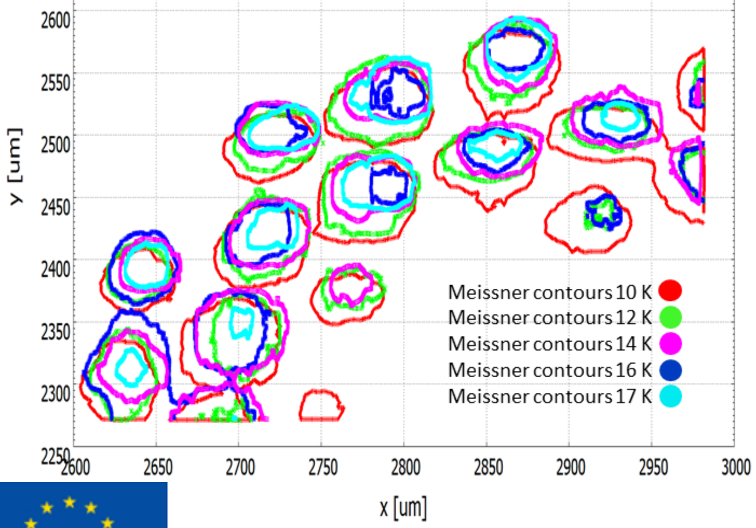
$$F_p = \eta L f_p = -\eta L \Delta W / x$$

$F_{p,max\ (model)}$=16.8% $F_{p,max\ (exp.)}$

[4]Dew Hughes, D. (1974). Flux pinning mechanisms in type II superconductors. *Phil.Mag.*

Experimental results: radial inhomogeneities





Assessing T_c distribution ≡ radial A-15 inhomogeneities (coarse to fine grain size region). AC-susceptibility method (SQUID) was used to identify the Meissner *shielding contours*

With T_{sample} increasing, a shrink of the Meissner shielding volumes is expected

Ingredients:

- SQUID or Scanning Hall probe microscopy (SHPM) → ✓
- $B_{app}<B_{c1}$ (Meissner state) → 30 µT
- No other magnetic signals → ✓
- Temperature sweep → 5 to 20K
- Thin and flat sample for SHPM → Best achievement: Thickness= 15 µm Flatness= 7%

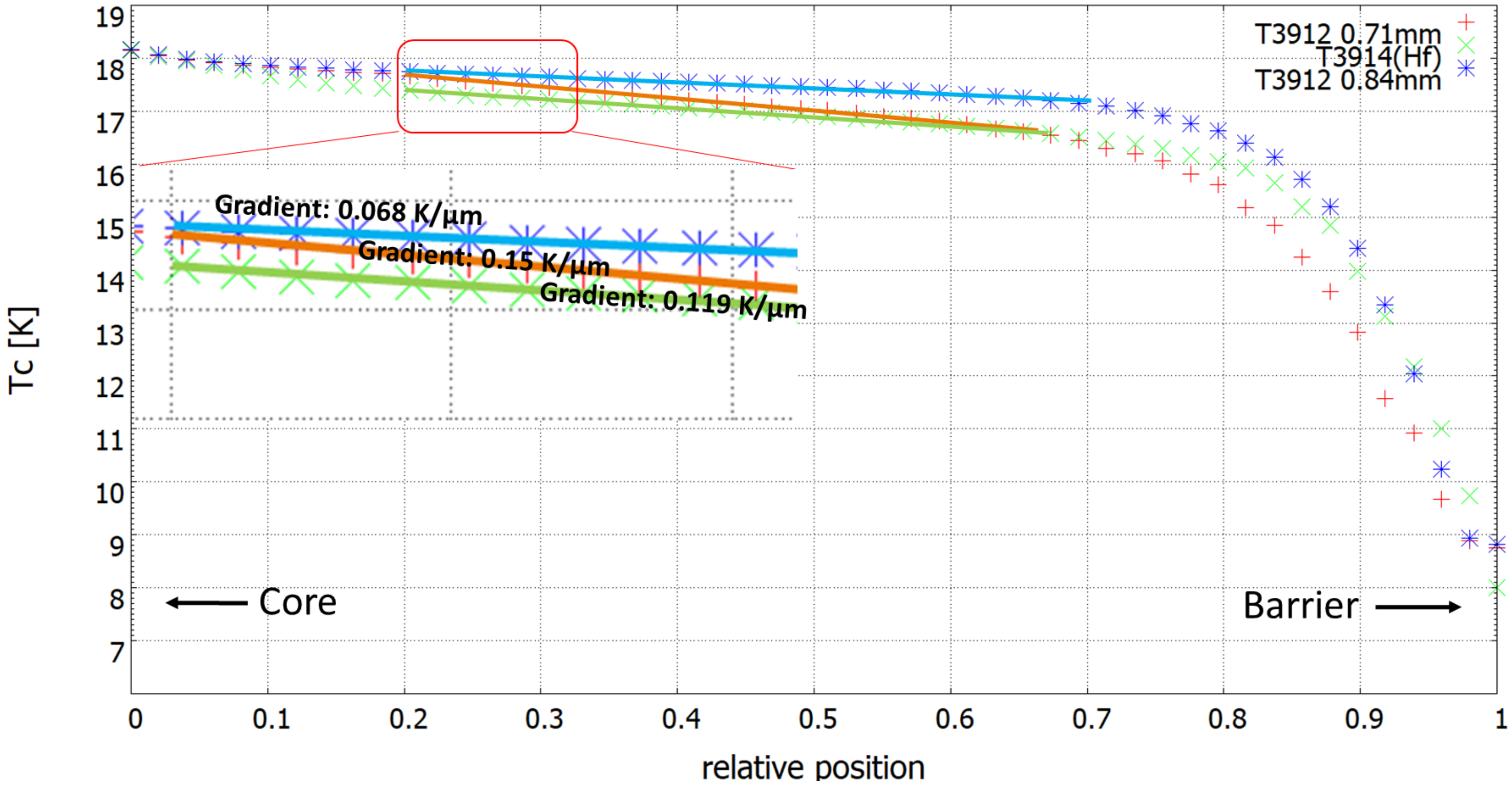
→ Radii(T) of the single sub-elements are then converted to relative position inside the inner and outer radius of the A-15 region

Experimental results: radial inhomogeneities

Evaluation [5]model based on some assumptions:

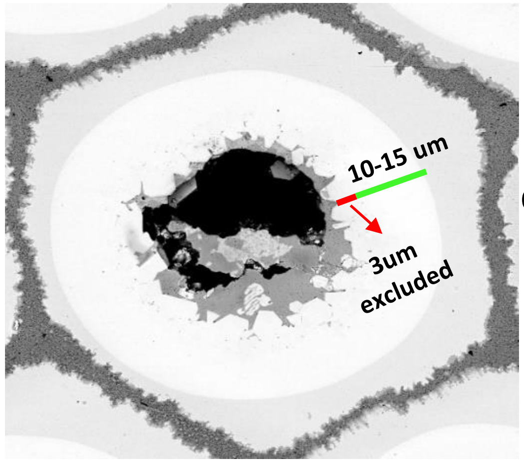
[5]T.Baumgartner et al., Supercond. Sci. Technol. 30 (2017) 014011

1. Sub-elements inside the sample are parallel tubes with circular cross sections;
2. All sub-elements are identical (geometry/composition);
3. Each sub-element exhibits a monotonic radial Sn gradient with the highest value on the inside



Simulation runs on a single sub-element by changing its radial T_c distribution until the computed $m(T) \equiv m_{exp}(T)/N$

Experimental results: radial inhomogeneities



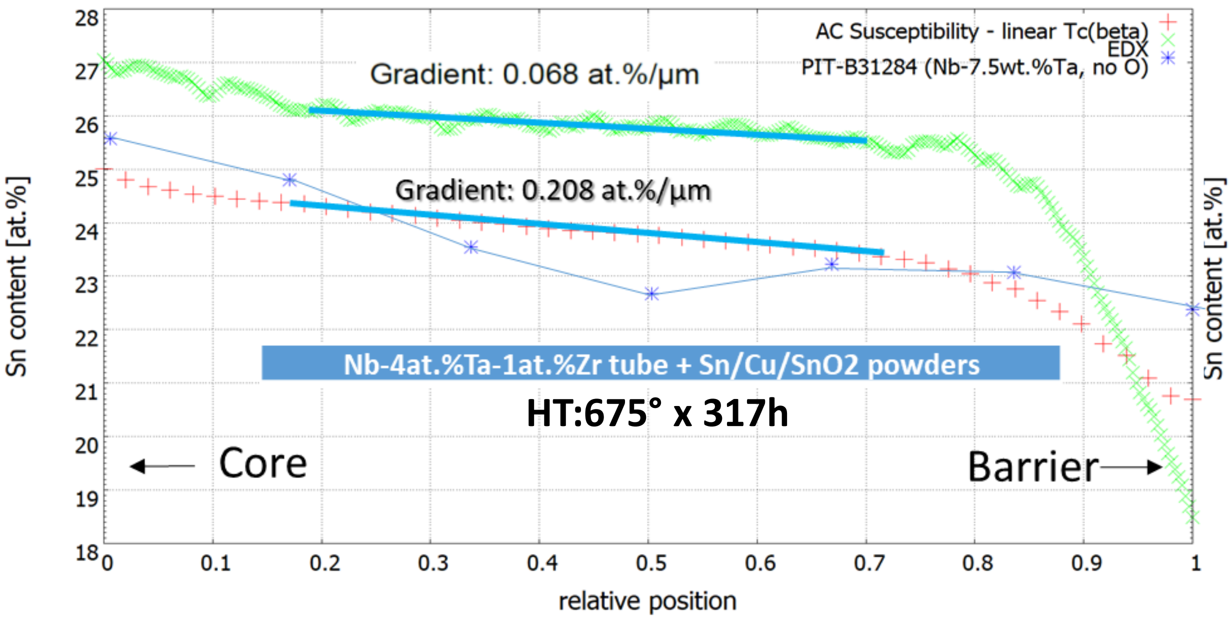
Radial inhomogeneitites → radial Sn content:

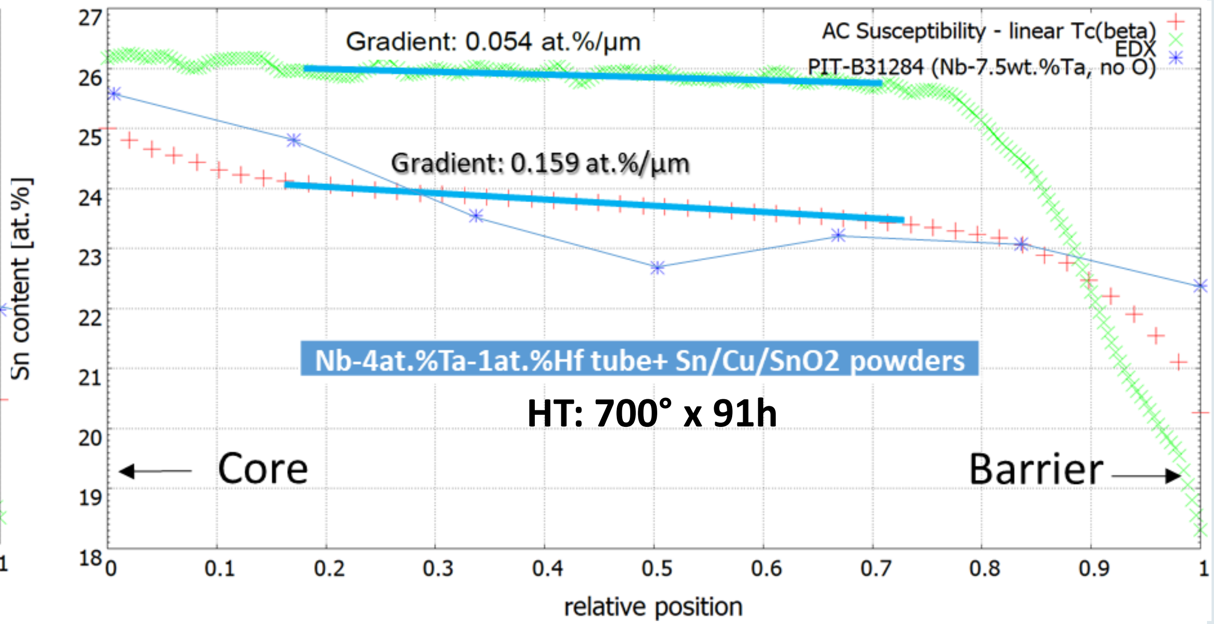
For a more accurate analysis, the effective boundaries of the single sub-elements A-15 edges were evaluated by means of pixel counting

[5]Godeke's T_c (β) has been used in order to relate the T_c distribution to the at.% of Sn

[5]A.Godeke, Supercond. Sci. Technol. **19** (2006)

$$Tc(\beta) = \frac{Tc_{min} - T_{cMAX}}{1 + e^{(\beta - \beta_0)}} + T_{cMAX}$$

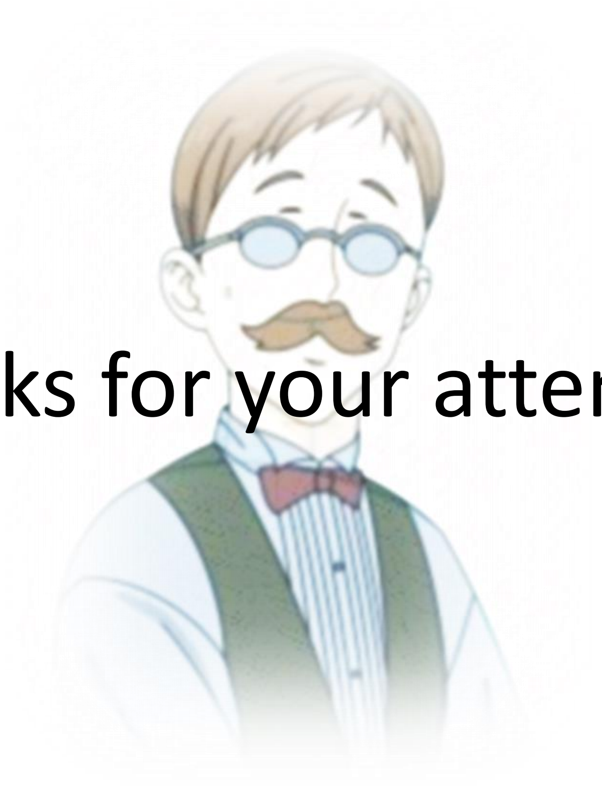




EDX and magnetic evaluations show similar behaviour but different absolute values

Conclusions

- APC-Nb_3Sn wires produced with 4at.%Ta additions confirm their high J_c achievements (beyond FCC-goals), as well by means of magnetometry;
- 1at.%Hf+O doped sample shows similar performances if compared with the 1at.%Zr-doped ones (even better homogeneity);
- Peak shifts in f_p show a possible point pinning contribution: a further investigation is needed;
- Microstructure has not changed in ternary compounds: same grain and nano-particle size as in the binary generation, still no preferential deposition;
- Inhomogeneities: more accurate investigation of the model (inter-granular gradient to be raised/lowered) or of the T_c-Sn% (still referring to binary compounds) is needed;
- Further T_c distribution analysis with SHPM coming: difficult to perform but with less restrictions than AC-susceptibility.

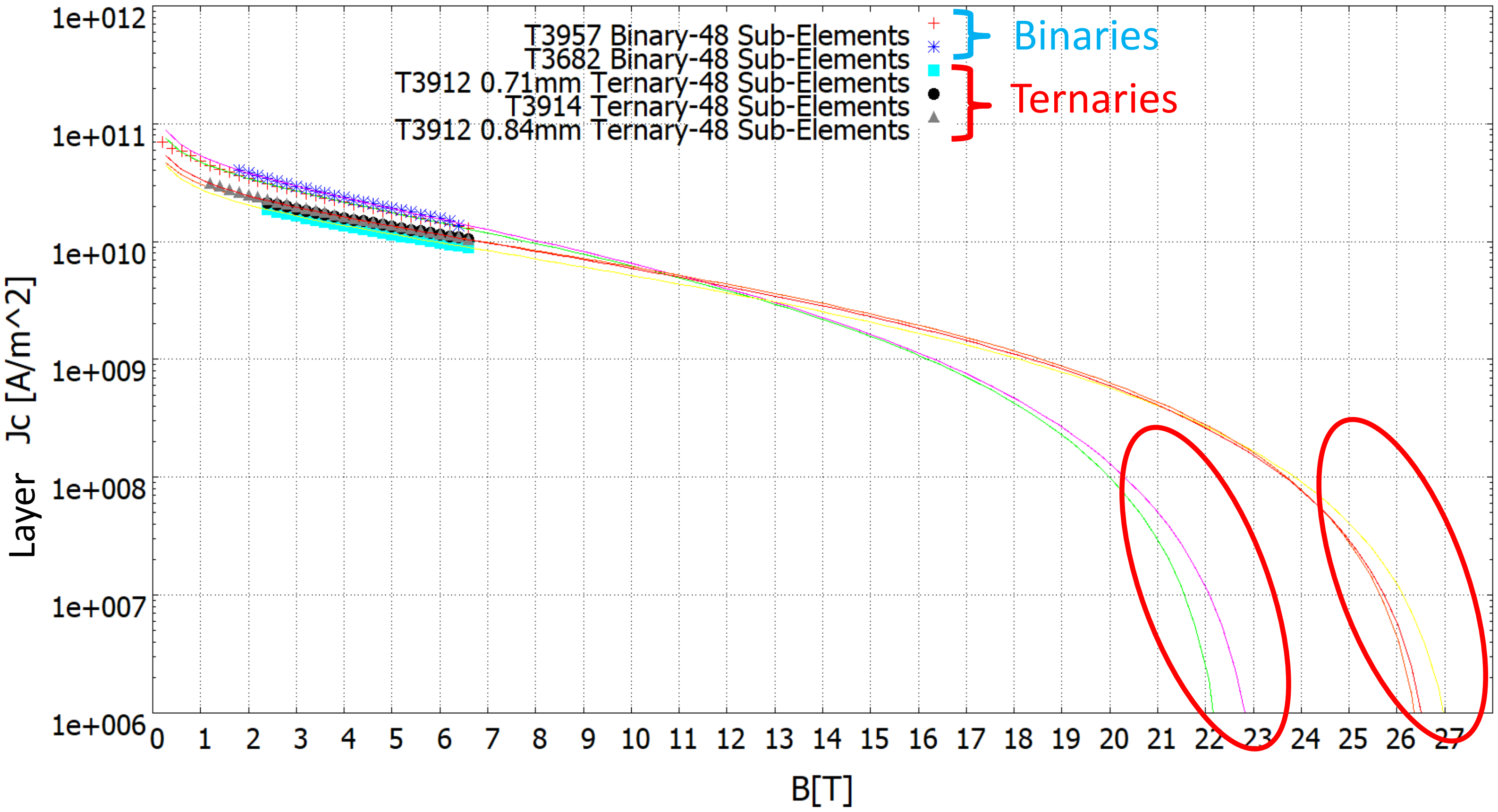


Thanks for your attention!

Backup

Sample	B_{c2avg}	p_{avg}	q_{avg}
T3657 (binary)	22.6	0.71	2.19
T3682 (binary)	23.4	0.64	2.15

Sample	B_{c2avg}	p_{avg}	q_{avg}
T3912 d=071	27.3	0.68	2.28
T3914 (Hf-sample)	26.8	0.73	2.29
T3912 d=0.84mm	27.8	0.68	2.32

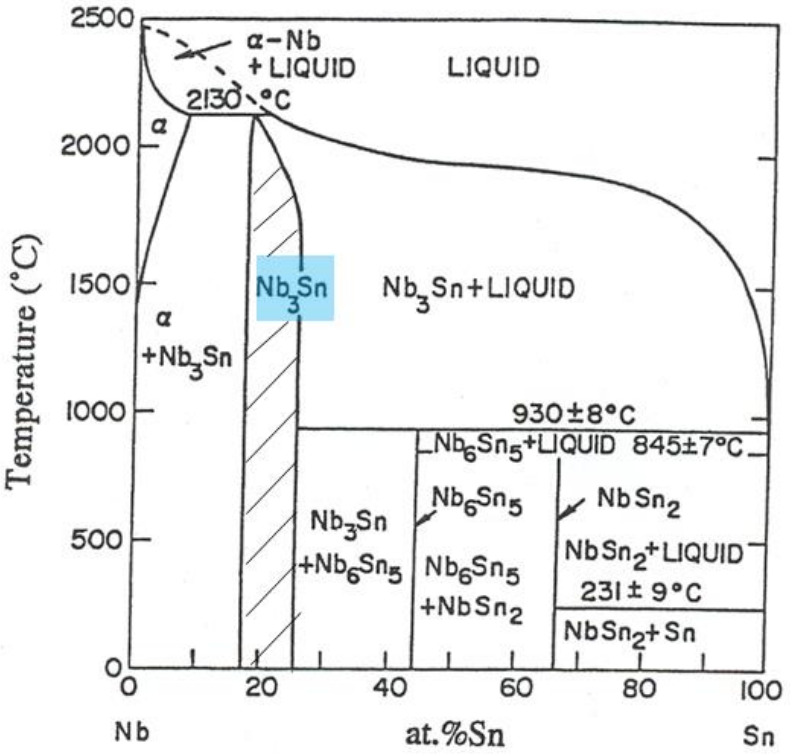


$$Fp(B) = Fpmax * C * \left(\frac{B}{B_{c2}}\right)^p * \left(1 - \left(\frac{B}{B_{c2}}\right)\right)^q$$

- Elementary pinning force approach:

$$f_{p,max} \cdot \rho_{defects}$$

$$f_{p,max} = \frac{U_{p,max}}{\xi} = \frac{\mu_0 H_c^2}{2}\frac{4}{3}\pi r_p^3$$



- [4]Dew Hughes approach:

$$F_p = \eta L f_p = -\eta L \Delta W / x$$

$$F_p(B) = \frac{\frac{BV_f}{\Phi_0} \cdot \frac{\pi\xi^2 (H_{c2} - H)^2}{4.64k^2}}{\frac{r_p}{2}}$$