Optimization of the Heat Treatment schedule for NED PIT Nb_3Sn strand

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Introduction: NED specifications

NED main goal- to design and build Nb_3Sn high-field magnet but mainly limited to conductor due to financial constraints.

Main specifications for NED strand

Diameter	**1.250 mm,**
• **Eff. filament diameter**	**< 50 µm,**
• Cu-to-non-Cu ratio	1.25 ± 0.10,
• Filament twist pitch	30 mm,
• **non-Cu J_C *(target)***	**1500 A/mm² @4.2 K & 15 T,**
• **minimum critical current**	**1636 A at 12 T,**
(target)	**818 A at 15 T,**
• RRR (after heat treatment)	> 200.

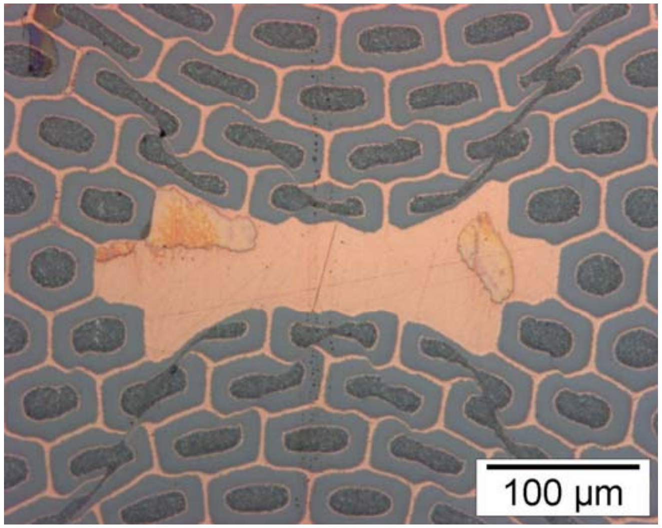
Two orders were placed in 2004 to ***Alstom-MSA*** **(IT)** and ***SMI*** **(PIT)**

R&D phase: B215 strand development

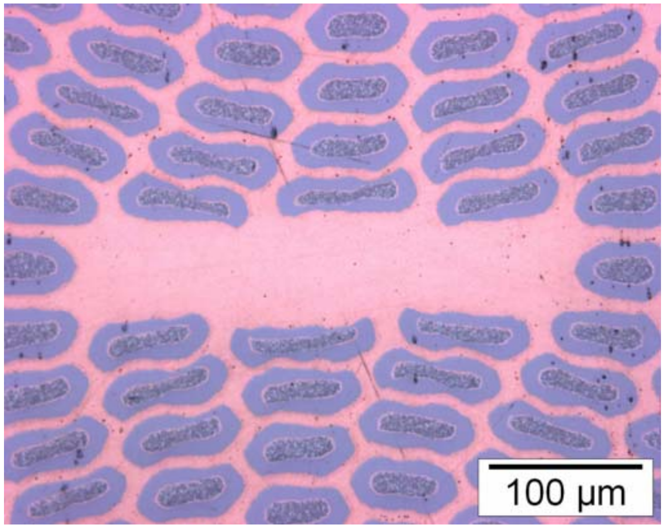
- During R&D phase, SMI developed ***successfully*** a strand, B215, including 288 filaments instead of 192 previously (B179 strand).

- The strand design incorporated more copper between the filaments (tubes) for an improved mechanical behavior following cabling deformation:

Flat rolling deformation

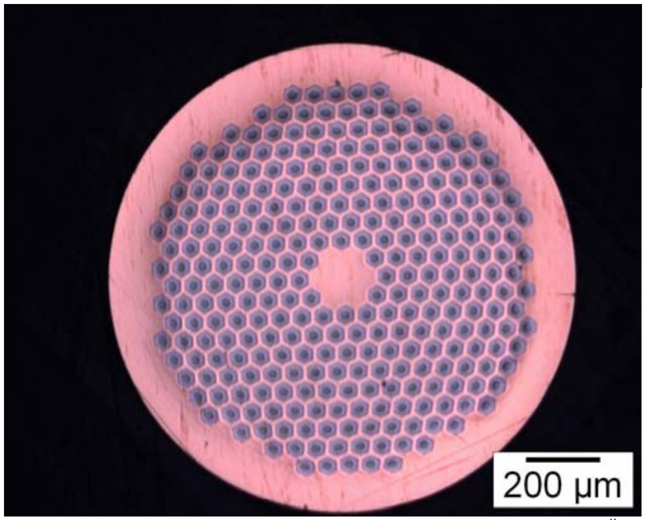


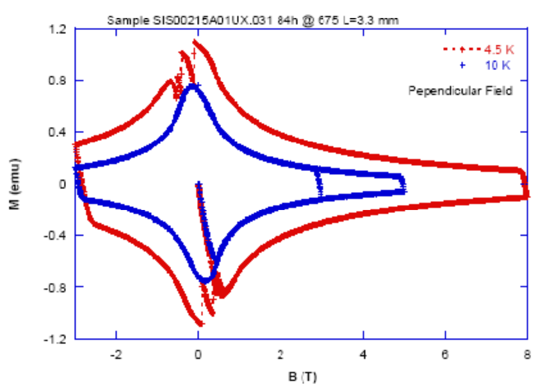
B179 (25 % def.)



B215 (27 % def.)

B215 strand characterization (standard HT)





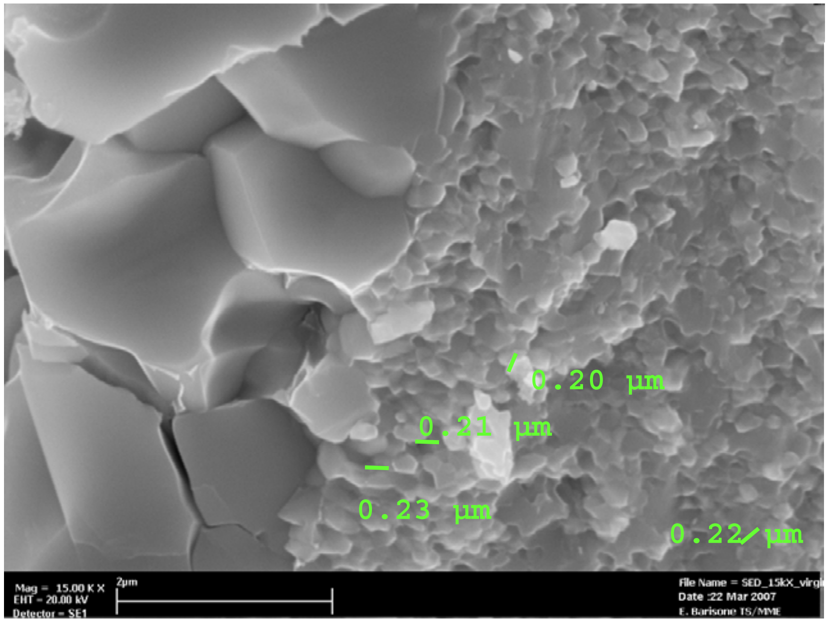
- 950 m produced in a lone length.
- 288 filaments (~ 50 µm), adequate Cu/non-Cu (~ 1.22), D_{st} = 1.257 mm.
- Max. measured ***J_c* ~ 2500 A/mm², *I_c*=1397 A** (Twente), 12 T and 4.2 K, 84 h @ 675 °C (provided by SMI).
- **I_c=756 A** @ 15 T, 4.2 K (only 8 % below spec.), **J_c=1350 A/mm²** (Geneva).
- RRR ~ 70-80 (virgin samples).
- Magnetization meas.: ***few flux jumps***.

HT optimization studies on B215: motivation

- In PIT reacted strands: A15 phase composed of big grains (**1-2 µm**, **~ 30 %**) and small grains (~ 200 nm). J_c contribution of big grains **negligible**.

- The idea: try to improve I_c and RRR by reaction temperature decrease in order to (tentatively):

1. To reduce small Nb_3Sn grain size
2. To reduce big grain area (***possible?***)



B215 sample, 84 h @ 675 °C

HT optimization studies on B215: method

• Due to limited amounts of available B215 strand and to limited reaction oven resources , a systematic optimization study with broad scanning of treatment temperature and duration ***not possible***.

• Instead, a kind of ***"trial and error"*** method used: a specific HT schedule is selected, then I_c, RRR measured, cross-section examined at microscope to evaluate the extent of the reacted area. According to these results, the next schedule is chosen…

• Various temperatures and durations already tried with the same temperature ramp of 50 ^{o}C/hour:

660 ^{o}C: 84 h

650 ^{o}C: 84 h, 120 h

625 ^{o}C: 200 h, 260 h and 320 h

Prominent results

120 h @ 650 °C

- I_c measurements performed at CERN and Twente. Consistent I_c data measured with maximal value of **1410 A** (12 T, 4.2 K), corresponding to $\mathbf{J_c \sim 2520\ A/mm^2}$, ~ 1 % larger than 84 h @ 675 °C.

- Although slight increase not significant, measurements more reproducible (I_c scatter of less than 3 % versus ~ 6 % for standard HT).

- Measurements at Geneva: 1 % as well at 15 T and 4.2 K, B_{c2}^K~25.6 T.

- B215 strand cabled at LBNL (summer 2007). Extracted wires treated 120 h @ 650 °C measured (CERN and Twente) to assess cabling degradation: reasonable degradation of 4-8 % consistently observed, as compared to 10-12 % degradation (LASA/Milan) for 84 h @ 675 °C.

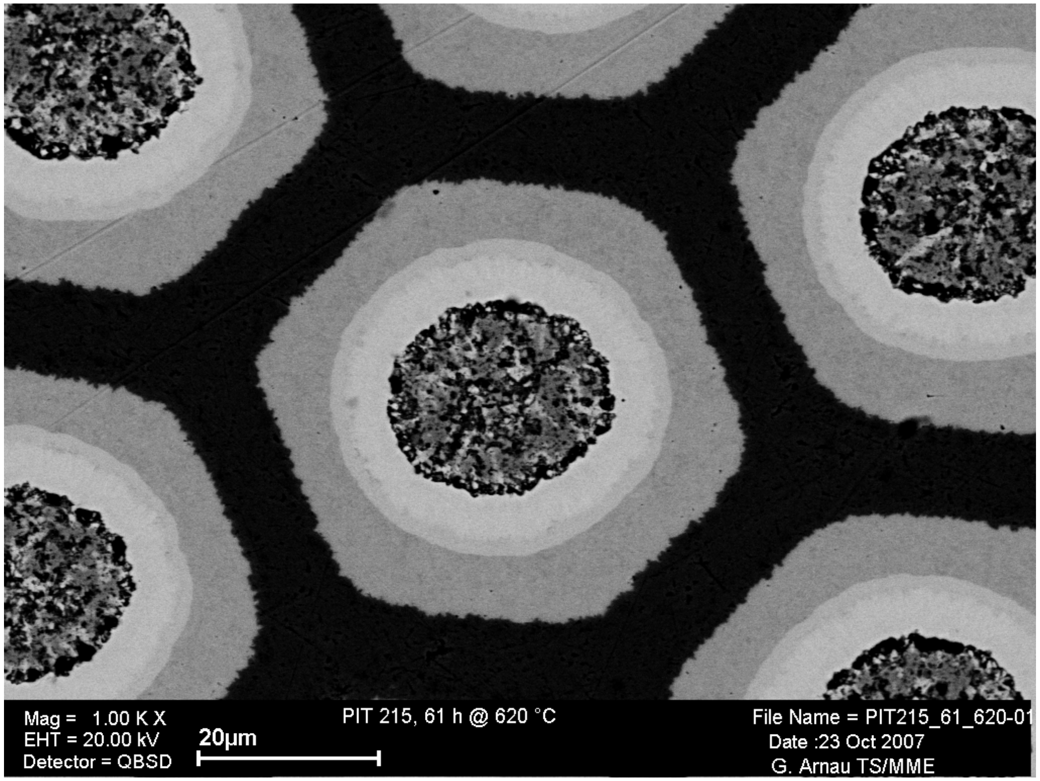
120 h @ 650 ^{o}C (end)

- From RRR point of view, 120 h @ 650 ^{o}C appears to be more favorable than 84 h @ 675 ^{o}C.
- Indeed, for virgin strands, RRR values, generally larger than 100 (max. 143) for 120 h @ 650 ^{o}C as compared to values not exceeding 80 (standard HT schedule).
- For extracted strands, samples at 120 h @ 650 ^{o}C have RRR ~ 100, comparable to virgin strands versus RRR=30-60 (84 h @ 675 ^{o}C)

320 h @ 625 ºC

After treatments at 650 ºC, decided to try 625 ºC.

As preliminary test, B215 sample treated 61 h @ 620 ºC to check reaction feasibility at such low temperature.



Courtesy of G. Arnau and C. Scheuerlein

- ***Good news***: thin **A15** layer already reacted (dark grey).
- ***Bad news***: large area of **Nb_6Sn_5** (white layer close to core) also present, to be converted to Nb_3Sn big grains (at least 25 % of reacted Nb_3Sn phase).

320 h @ 625 ^{o}C (continuation)

- After two trials at 625 ^{o}C (200 and 260 hours), 2 samples were treated 320 h @ 625 ^{o}C at CERN.

- I_c data found consistent within 0.2 % for both samples with impressive **I_c = 1496 A** @ 12 T and 4.22 K, corresponding to ***J_c = 2665 A/mm^2***, **+ 7 %** as compared to standard HT, $B_{c2}{}^{K}$ ~ 27 T (to be confirmed).

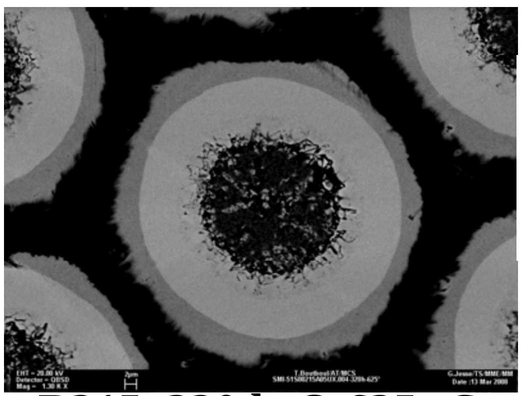
- ***New record!!*** Measurements currently underway at Nijmegen (Netherlands) on virgin and extracted wires to confirm these results, to measure at higher fields and assess cabling degradation.

- RRR data impressive as well since ***RRR ~ 220*** for virgin strands!!

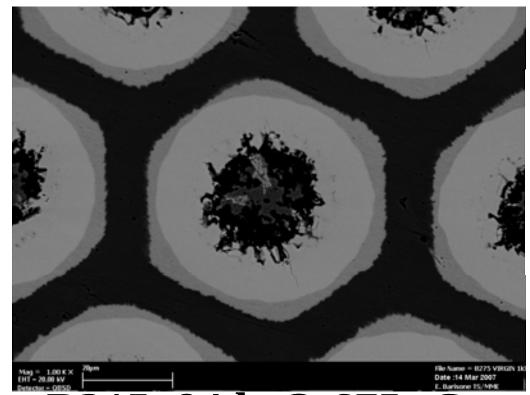
SEM examinations

SEM examinations performed at CERN on B215 samples reacted either 320 h @ 625 ºC or 84 h @ 675 ºC to explain J_c enhancement.

1. Filament reaction rate (on polished samples)



B215, 320 h @ 625 ºC



B215, 84 h @ 675 ºC

Un-reacted barrier:

25 % of filament (320 h@625 ºC) versus ***23 %*** (84 h@675 ºC)

Despite higher I_c, 320 h@625 ºC sample slightly **less reacted**!!!

Thus less Nb_3Sn for 320 h @ 625 °C but "higher quality"?

To be checked:

2. Sn content in reacted Nb_3Sn

- SEM/EDS analyses done on polished samples.

- 320h @ 625 °C sample (fine grains) contains **stochiometric** content: ***~ 24.7 at. % Sn*** in average as compared to ***~ 24.2 at. %*** (standard HT).

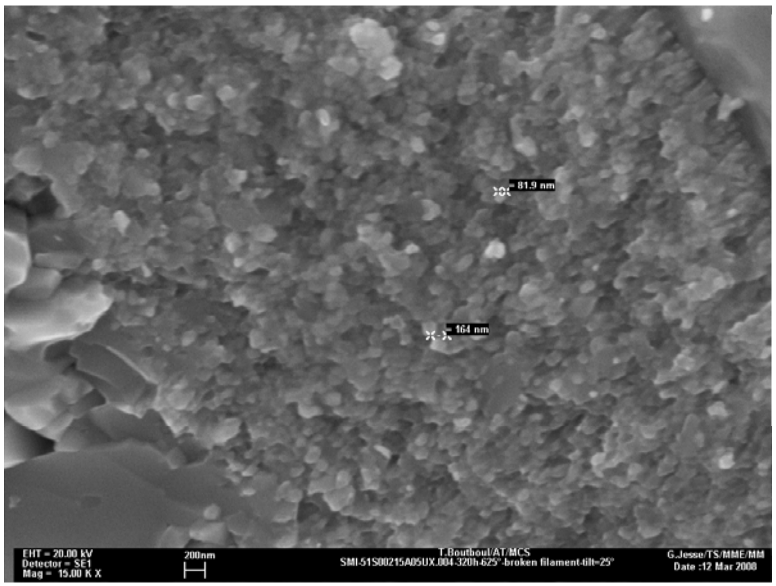
3. Coarse grains versus fine grains

- Ratio of big grains in Nb_3Sn phase estimated from fractured samples.

- ***~ 30 %*** of A15 area composed of coarse grains not contributing to J_c for both 320 h @ 625 °C (29 %) and 84 h @ 675 °C (31 %).

- Difference not significant. Confirmation, after 61 h @ 620 °C trial, that **difficult** to **reduce big grain** area ratio.

4. Fine grain size



B215, 320 h @ 625 ºC

- SEM fractographs used to estimate fine grain dimensions. SEM not convenient tool for this task (TEM more suitable) but large statistics used.
- Semi-quantitative evaluations good enough at least for comparisons.
- Effective grain diameter estimated on basis of measured grain areas.

Mean fine grain size ***smaller*** for 320 h @ 625 ºC than standard HT:

~ 160 nm versus ***~ 180 nm***

Conclusions and perspectives

- During R&D phase for NED, SMI developed a strand with ~ 50 µm filament size and J_c ~ 2500 A/mm^2 at 12 T and 4.2 K with HT schedule recommended by firm (84 h @ 675 ^{o}C). Optimization study of HT launched at CERN to improve strand performance, with reaction temperature down to 625 ^{o}C and duration in 84-320 hours range.

- For 320 h @ 625 ^{o}C, ***increase of 7 %*** as compared to standard HT observed in critical current (***I_c ~ 1500 A*** @ 12 T and 4.2 K, ***J_c ~ 2670 A/mm^2***), **new record**!! RRR high (***~ 220***) as well for this treatment.

- SEM examinations: 320 h @ 625 ^{o}C sample has slightly less reacted Nb_3Sn than standard HT sample but ***higher quality A15 phase***:

1. **higher Sn** content (***24.7 at. %*** vs ***24.2 at. %***)
2. **smaller grain size** (***160 nm*** vs ***180 nm***)

Conclusions and perspectives (end)

• SEM examinations showed, as well, that reducing temperature did not reduce coarse grain area. This 1-2 µm grain region poorly contributing to J_c could be unavoidable ***(~ 30 %*** of total reacted Nb_3Sn).

• Measurements currently underway at Nijmegen to confirm nice results and assess cabling degradation on both I_c and RRR. HT schedule at 625 °C but longer than 320 h should be shortly launched at CERN to assess J_c limits of this strand.

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