# Deformation Behaviour and Cabling Degradation of $Nb_3Sn$ Wires

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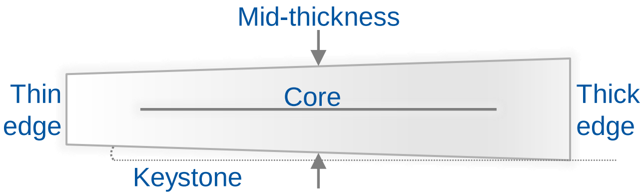
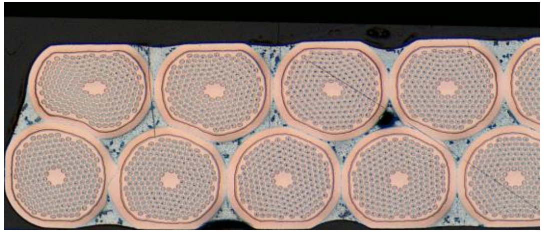
# Introduction

- For accelerator applications, $Nb_3Sn$ wires are assembled in the form of Rutherford cables, applying significant deformation to the wire, especially at the cable edges
- The resulting distortion of the wire cross-section before heat treatment can impact the $I_c$, RRR and stability:
  - Effects depend on the wire design, cabling and heat treatment parameters, and can vary between spools/billets
  - Important to assess the behaviour both for each type of wire and for individual billets
- As cable production consumes significant wire lengths and time, uniaxial rolling is commonly used as an (imperfect) proxy for initial acceptance tests
- Extensive data from systematic testing are available from the Bruker RRP® and PIT wire designs procured at scale for magnet production
- There is also a need to characterise a broader range of wire designs in the context of the High Field Magnets (HFM) programme:
  - Different RRP® layouts, especially at larger diameter
  - Wires developed in collaborations with other manufacturers: notably distributed tin (DT) wire from JASTEC and KAT
  - Ultimately, wires developed towards the challenging performance targets of ultimate $Nb_3Sn$ dipole magnets, e.g. a non-Cu $J_c$ of 1500 A/mm$^2$ (4.2 K 16 T)
- This presentation summarises the rolling and cabling behaviour of several wire types, combining rolling studies of individual spools with statistical analysis of rolling and cabling degradation over large-scale procurement

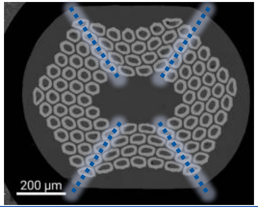
# Rutherford Cable Deformation

- Strands at the centre of the cable width typically have a **nominal** thickness reduction ~11 %
  - Rolling studies typically performed for 10 % and/or 15 % uniaxial rolling reduction
- The real deformation, especially at the (thin) edge, is more severe and not uniaxial
  - All strands experience this periodically, at a transposition length typically much shorter than the lengths of samples used for $I_c$ and RRR
- Stresses align along shear planes, but the resulting deformation of sub-elements depends on the wire type and its local orientation [1]
- Sub-element deformation affects performance via several mechanisms:
  - Sub-element shearing and merging
  - Changes in local barrier thickness and diffusion distances



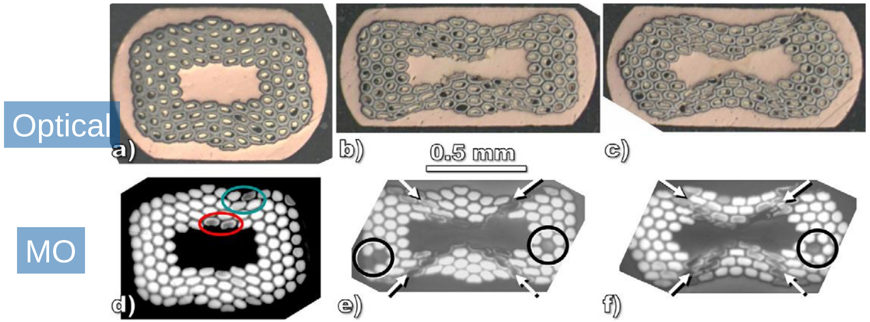



Courtesy of Algirdas Baskys
[1] *IEEE Trans Appl. Supercond.* **33** (5) 4801605, 10.1109/TASC.2023.3265910

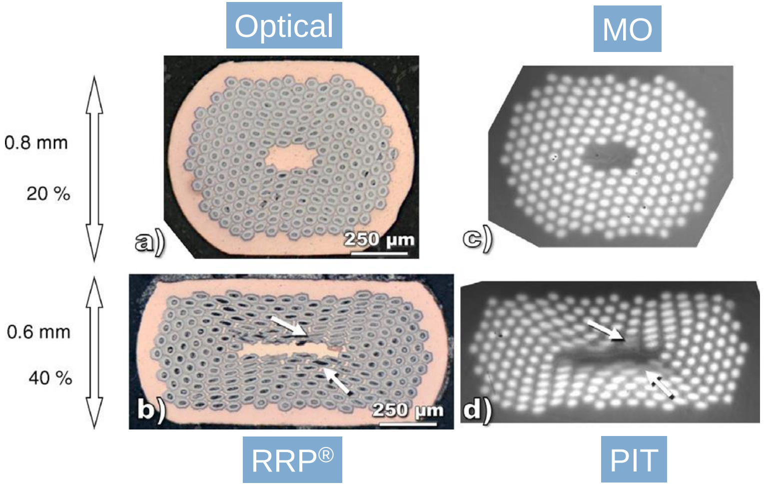


# Previous Work

- The rolling and cabling degradation of RRP® and PIT wires has been extensively studied over many years, especially for wires of interest for HL-LHC and LARP
  - The key deformation characteristics were imaged, and their impact on superconducting performance directly observed, many years ago

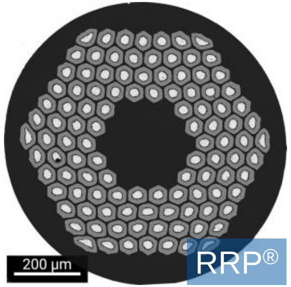


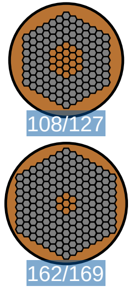

A A Polyanskii et al., *Supercond. Sci. Technol.* **22** 095008 (2009)

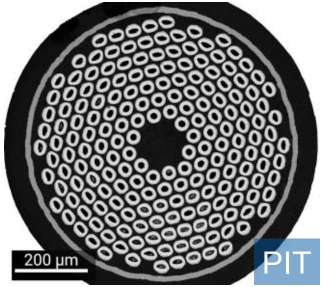


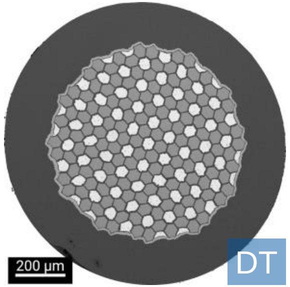
# Wire Types

- Three wire types of interest:
  - RRP® (Bruker OST) – internal tin sub-elements in a distributed barrier configuration
  - PIT (Bruker EAS) – powder-in-tube filaments, with an additional common diffusion barrier in the ‘bundle-barrier’ (HEP) variant (‘PIT-BB’)
  - Distributed tin (JASTEC, KAT) – internal tin wire with alternating Nb and Sn(Ti) modules and a common external diffusion barrier
    - R&D wires developed in collaborations in the context of the High Field Magnets programme
- Each type has a distinct deformation behaviour and would be expected to show a different sensitivity of $I_c$ and RRR to deformation
  - For RRP®, different layouts/restacks can be compared
  - For RRP® and PIT, characterisation of large procured quantities permits statistical analysis

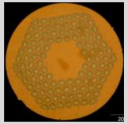
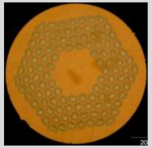
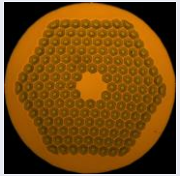
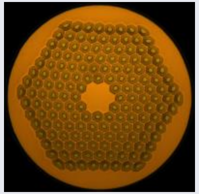


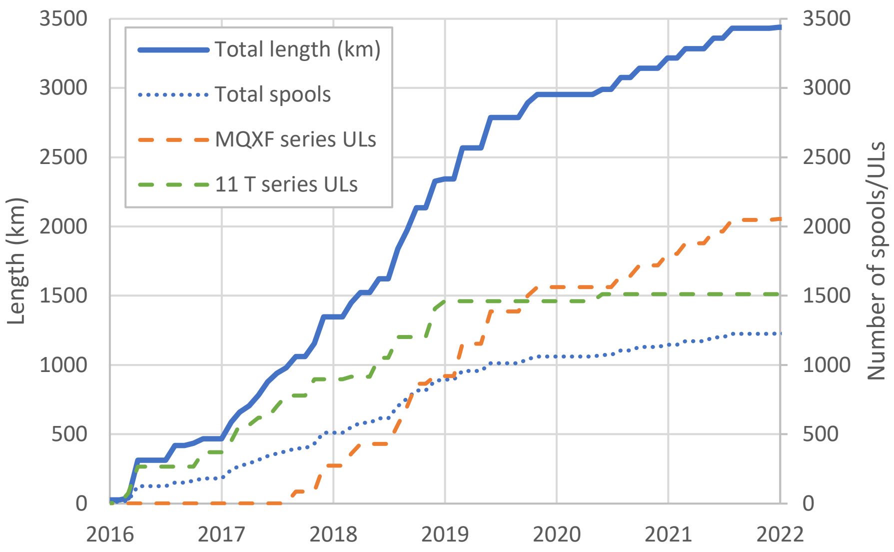



# RRP® Wire Characteristics

| | HL-LHC | | HFM | |
|---|---|---|---|---|
| | **11 T dipole** | **MQXF** | **ERMC-1** | **DEM-1.1** |
| |  |  |  |  |
| **Diameter (mm)** | 0.7 | 0.85 | 1.0 | 1.1 |
| **Layout** | 108/127 | | 162/169 | |
| **$d_s$ (μm)** | 45 | 54 | 58 | 64 |
| **Cu/non-Cu** | 1.15 ± 0.1 | 1.2 ± 0.1 | 0.9 ± 0.2 | |
| **Nb:Sn** | 3.6 (reduced Sn) | | 3.4 (standard Sn) | |
| **Heat treatment** | 650 °C 50 h | 665 °C 50 h | 650 °C 50 h | 665 °C 50 h |

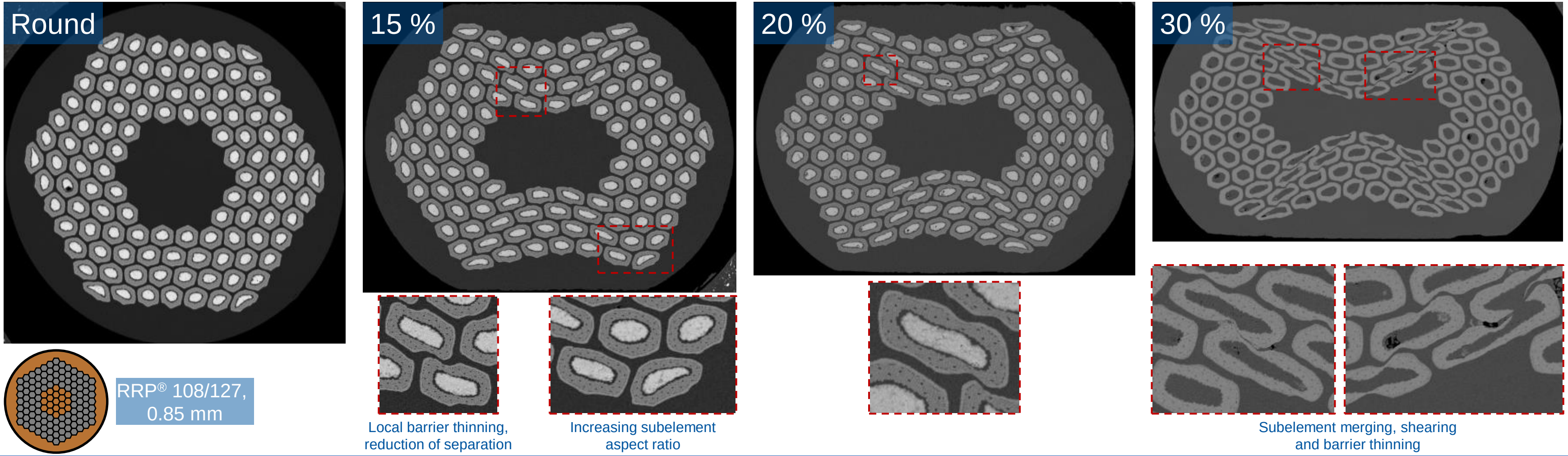
# RRP®: Statistics from Acceptance Tests

- For HL-LHC, 108/127 RRP® was procured in significantly quantities at 0.7 mm (for 11 T dipoles) and 0.85 mm (for MQXF) diameter
  - Acceptance tests included measurements of every spool
  - 15 % rolling data for all billets
- Similar testing, but with a lower overall quantity and sampling rate, applied to 162/169 RRP® wire procured for FCC/HFM
- The following slides include both:
  - Studies at different rolling reductions for individual spools
  - Statistical evaluation of the behaviour over all procured series spools
- In each case, data are presented only for the standard heat treatment



# RRP® Rolling: Key Features

- Visually apparent that progressive rolling:
  - Increases subelement aspect ratios
  - Locally reduces diffusion barrier (and Nb filament pack) thickness
  - Shears or merges adjacent subelements
- These observations can be quantified by image analysis:
  - Performed for a set of adjacent samples from **one spool** reacted in a single heat treatment

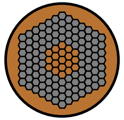



Local barrier thinning, reduction of separation

Increasing subelement aspect ratio

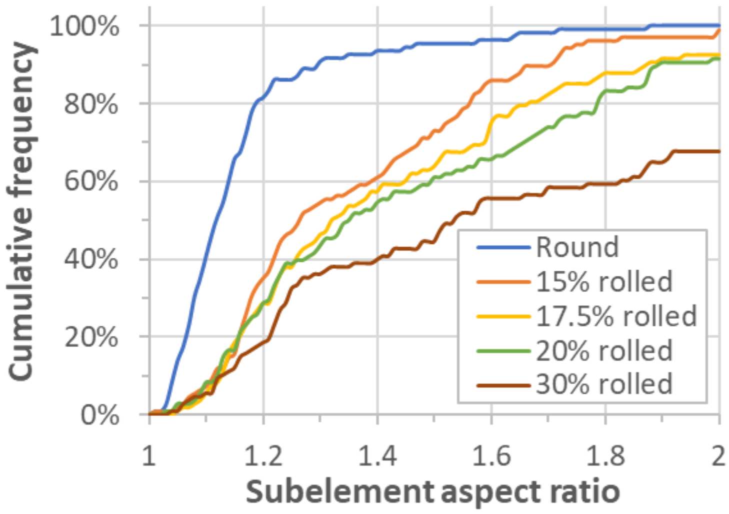
Subelement merging, shearing and barrier thinning

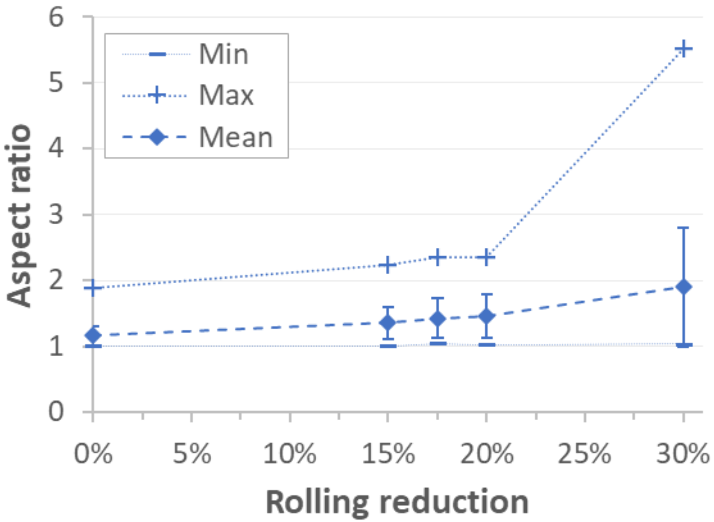
# RRP® 108/127: Aspect Ratio



RRP® 108/127, 0.85 mm

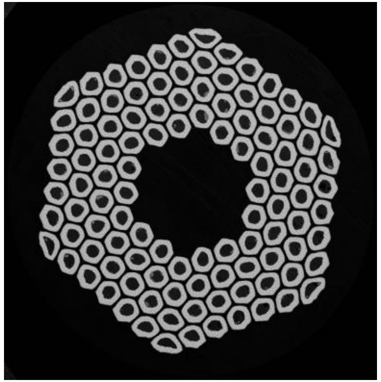
- The average subelement aspect ratio increases, and the distribution of aspect ratios broadens, with increasing rolling reduction
  - Particularly sharp increase from 20 % to 30 % rolling, corresponding to more frequent shearing/merging of subelements
  - Similar observations for subelement area and perimeter

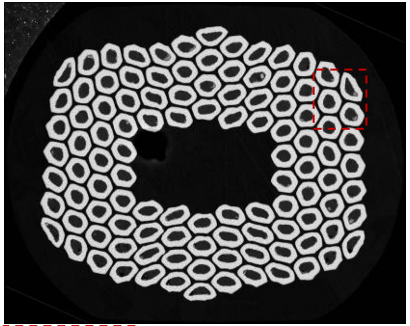


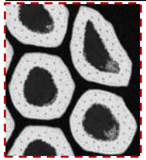



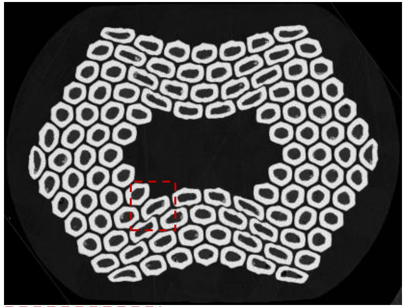
# RRP® Rolling: After Reaction

- After reaction, the aspect ratio distribution remains similar but areas expand (due to the expected volumetric expansion of Nb → $Nb_3Sn$)
- At 20 % and 30 % rolling, local instances of complete barrier reaction and/or breaks due to shearing become more prevalent, resulting in increased Sn loss to the matrix, and intermittent discontinuities and contacts in $Nb_3Sn$ subelements

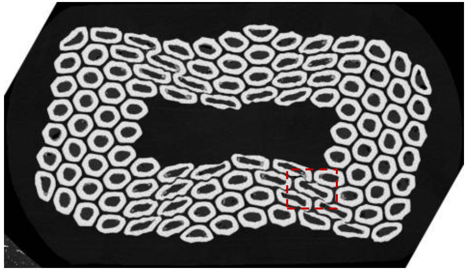


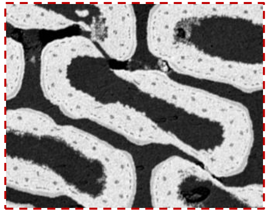






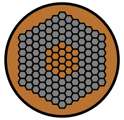
Locally complete barrier reaction, variable $Nb_3Sn$ thickness



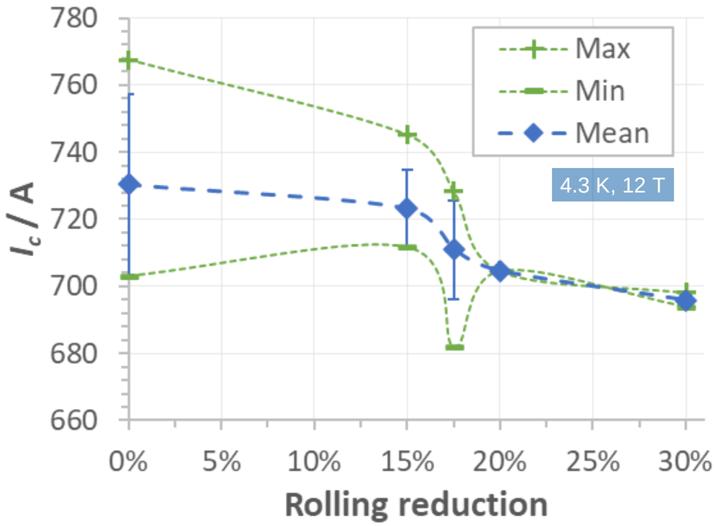


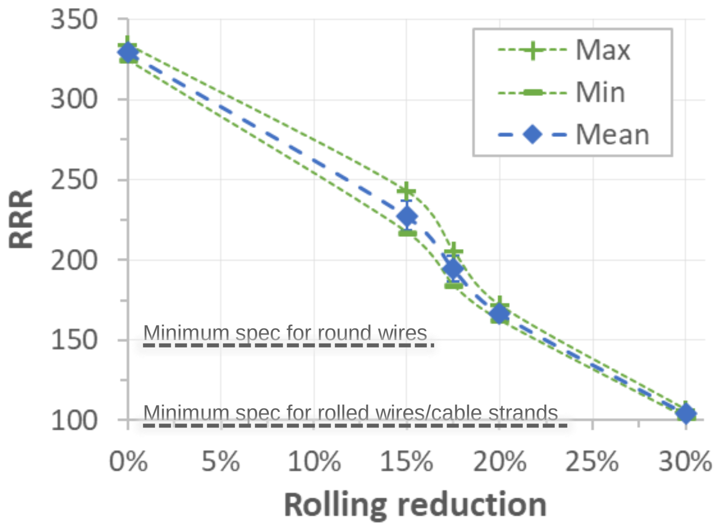
Contact between, and discontinuities in, $Nb_3Sn$ regions

# RRP®: Effect on $I_c$ and RRR



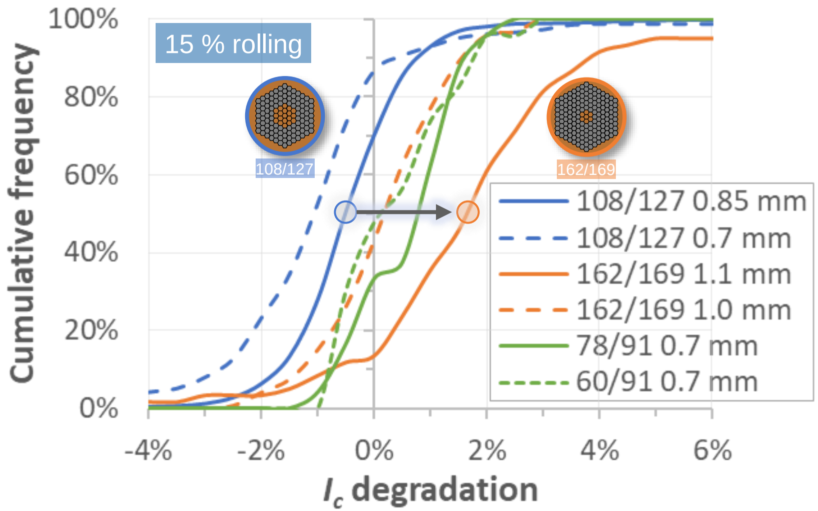

- $I_c$ and RRR both decrease progressively on rolling, on average, with a steeper gradient between 15 % and 20% rolling reduction
  - $I_c$ – significant spread at modest rolling reduction: average 'degradation' is *negative*, **-0.6 %**, for 15 % rolling
  - **RRR** – less variable, with a significant degradation even for small rolling reductions: average **36 %** for 15 % rolling

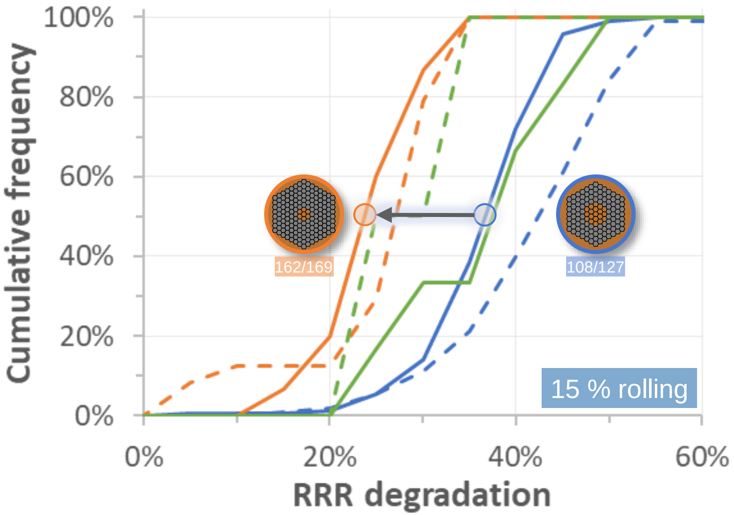


# Comparison of RRP® Layouts

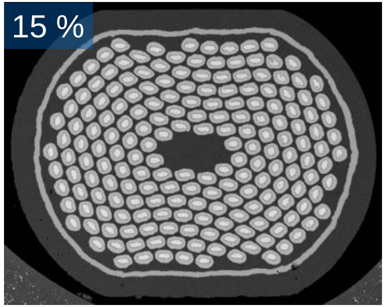
- For 15 % rolling, acceptance test statistics are available for a significant number of spools
- Relative to 108/127 at 0.85 mm, 162/169 at 1.1 mm shows (on average):
  - **Higher** $I_c$ degradation –**1.5 %,** cf. -0.6 %
  - **Lower** RRR degradation – **23 %**, cf. 36 %

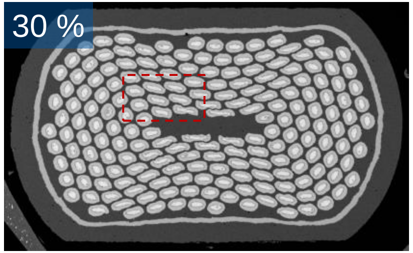


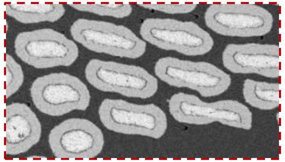



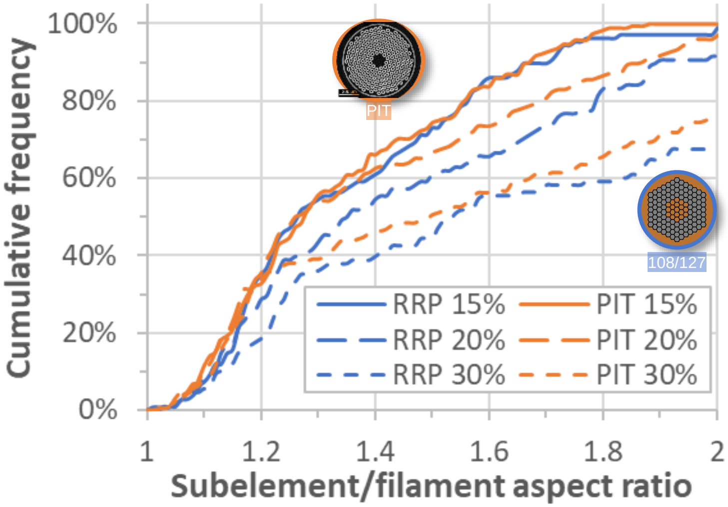
# Comparison of RRP® and PIT (1)

- The behaviour is similar for all tested RRP® wires
- Differences between (say) 108/127 and 162/169 may also be partly due to differing diameter, Cu/non-Cu, Sn stoichiometry, or HT cycles
  - For the same restack, $I_c$ degradation slightly higher for the larger-diameter wire
- **PIT** wires differ significantly in design (round filaments) and the nature of imperfections
- … but the evolution of the aspect ratio distribution of PIT and RRP® wires on rolling is remarkably similar



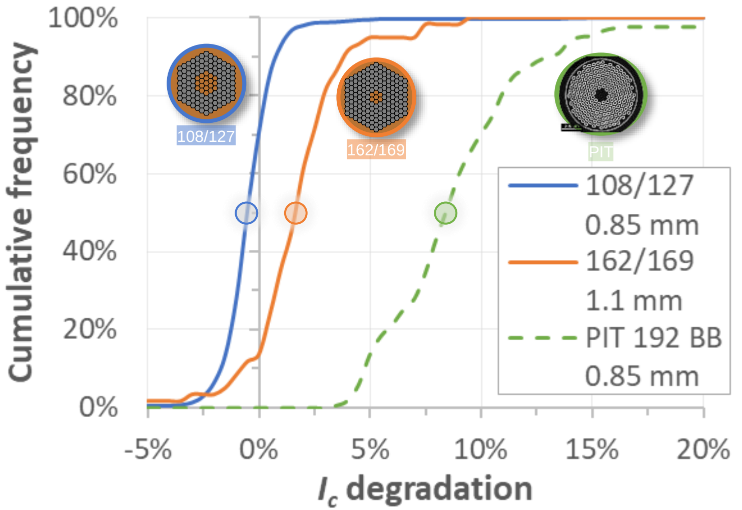






Cumulative aspect ratio distribution for 0.85 mm wires: RRP® 108/127 subelements and PIT-BB 192 filaments

# Comparison of RRP® and PIT (2)

- The performance impact of filament distortion for PIT BB does differ significantly
- Tin loss through locally fully-reacted or broken tubes:
  - Barely affects the measured RRR (due to the 'bundle barrier')
  - Significantly reduces $I_c$, as the reaction of the filament stops
- Consequently, PIT wire shows markedly higher average $I_c$ degradation (**mean 8.8 %**) on 15 % rolling, and a much broader distribution

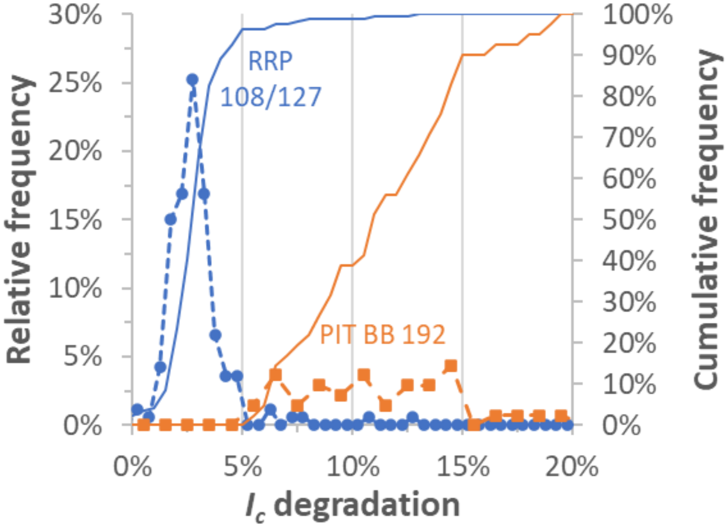



Comparison of $I_c$ degradation distributions after 15 % rolling for RRP® 108/127 and PIT-BB wires

# Comparison with Cables

- $I_c$ degradation on cabling depends on the cable configuration
- Taking statistics for cables of the HL-LHC MQXF design, using 0.85 mm strand, the **mean** $I_c$ degradation is:
  - RRP® 108/127: **2.8 %**
  - PIT BB: **11 %**
- …in both cases, **significantly higher** than for 15 % rolling:
  - For RRP, for the individual spool test, this is comparable to a rolling reduction of **17.5 %**
  - For PIT, the distribution of degradation values is too broad to compare with a single example
- RRR degradation is:
  - For RRP, **16.9 %** on average – approximately **half** that of 15 % rolled samples
  - Negligible for PIT due to the external barrier
  - Assessing degradation of RRR by rolling is **conservative**, as 15 % reduction is larger than experienced across the majority of the cable width
  - Local degradation at the cable edges should be separately measured *locally*

MQXF cable with 40 RRP® 108/127 strands)

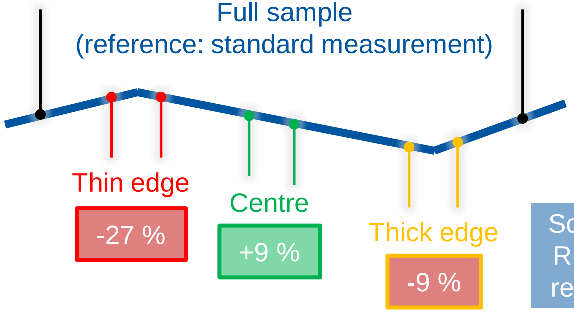


# Local RRR

- Typically, high purity (high RRR) Cu is used throughout the cross-section in manufacturing
- At the time of cabling, the wire is already heavily cold-worked from wire drawing
  - Little further change expected in Cu RRR or resistivity
  - Small differences across the cross-section may result from differences in starting Cu forms, grain sizes and wire processing routes etc.
- RRR is measured **after** heat treatment: variations are usually dominated by tin contamination
  - Longitudinally – for an extracted strand, the deformation locally at the cable edge reduces RRR
  - In the cross-section – RRR is often very high in regions protected by thick/intact diffusion barriers, but can be reduced strongly locally in interfilamentary regions

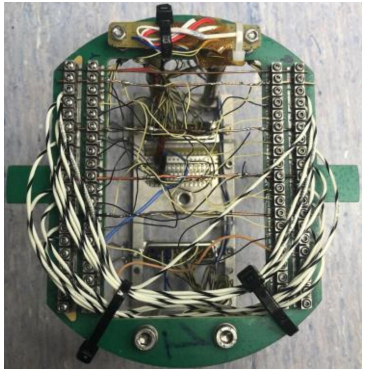
# Local RRR Degradation at Edges

- Local measurement of RRR confirms the expected behaviour
- Relative to the standard (for CERN) RRR measurement, RRR is:
  - Higher at the cable centre
  - Lower at the edges, especially the thin edge
- Values below are averages for two extracted strand samples of MQXF cables (RRP® wire)
- At present, samples may be bent for mounting in a sample holder optimised for straight samples: systematic measurement of local RRR will require a new design




Schematic of voltage taps for local RRR measurement, and example relative values for MQXF samples

RRR sample holder



# Local RRR Across Wire Cross-Sections (1)

- For wires with a common external barrier, etching the outer Cu allows the RRR to be measured for the central matrix only
  - For DT wires, this region is intentionally used for Sn transport, so it is **not** included in the Cu area for Cu/non-Cu
  - For PIT-BB, the barrier was added to retain acceptable RRR with a heat treatment achieving high $I_c$, so some RRR degradation of the matrix was anticipated, but internal Cu area **was** included in Cu/non-Cu
  - Further study is needed to assess the impact of RRR variations in the cross-section on stability, protection etc.

# Local RRR Across Wire Cross-Sections (2)

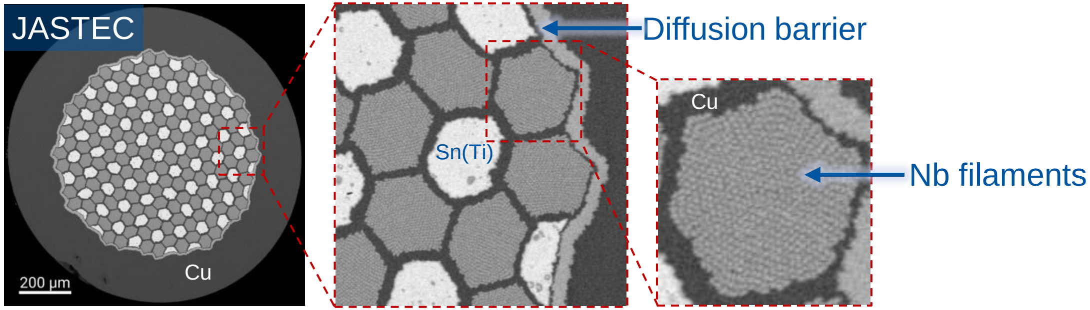
- The RRR of the central Cu of PIT-BB is very low, and reduced further on rolling
  - The RRR of the outer Cu alone is estimated to be very high, as for DT wires
- For a sample of wire with known low starting Cu RRR, the central Cu did not have a lower RRR after heat treatment than typical wires: the overall RRR is dominated by the outer Cu
- For a wire produced with improved filament geometry, the RRR was significantly improved, with the central region RRR (~140) potentially high enough to consider as part of the stabiliser

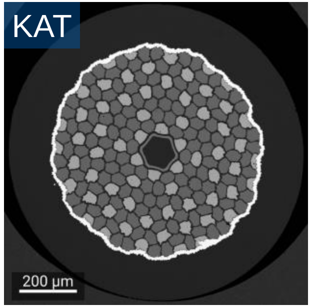
| PIT-BB Type | Sample | Rolling | RRR | Etched RRR (central Cu) | Estimated RRR of outer Cu |
|---|---|---|---|---|---|
| Series Wires | 1 | 0 % | 188 | 23.5 | 361 |
| | | 15 % | 189 | 7.7 | 353 |
| | 2 | 0 % | 134 | 11.5 | 319 |
| | | 15 % | 151 | 6.3 | 337 |
| Low Cu RRR | 3 | 0 % | 76.2 | 26.4 | - |
| Improved Geometry | 4 | 0 % | 250.1 | 139.8 | - |

RRR measurements courtesy of Al Baskys and Joanna Kuczynska

# Distributed Tin Wires

- Two manufacturers, JASTEC and KAT, have developed ‘distributed tin’ wires:



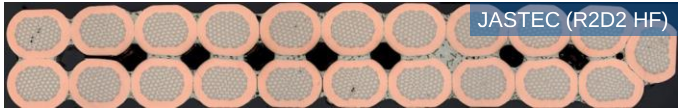

  - KAT’s designs have also included a copper core protected by an additional diffusion barrier

| Supplier | *d* (mm) | Cu/non-Cu | Nb/Sn modules | Mean piece length (m) |
|---|---|---|---|---|
| KAT | 1.0 | 0.93 | 138 + 54 | 1430 |
| JASTEC | 1.1 | 1.08 | 138 + 73 | 150 |

# Cabling Trials at CERN

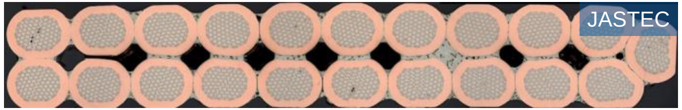
- Short trial cables successfully produced using cable designs established for magnet R&D activities
  - Rolling and cabling study for KAT previously reported at ASC 2022

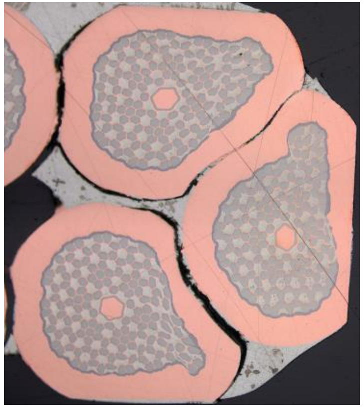
| Wire | | Cable | | | | | |
|---|---|---|---|---|---|---|---|
| Supplier | *d* (mm) | Layout | Strands | Key-stone | Width (mm) | Mid-thickness (mm) | Core |
| KAT | 1.0 | FalconD | 40 | 0.5° | 20.95 | 1.8 | 14×0.025 mm 316L |
| JASTEC | 1.1 | R2D2 HF | 21 | None | 12.579 | 1.969 | None |

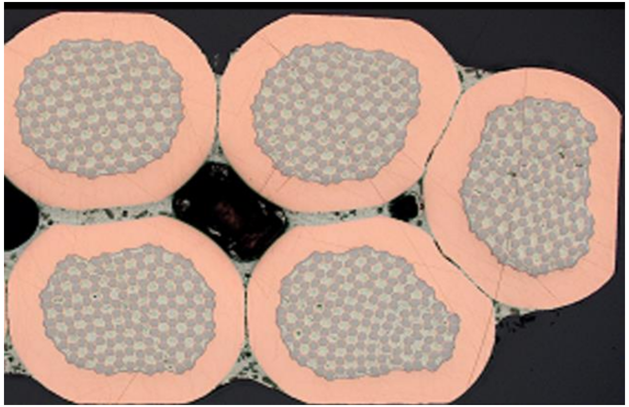



# Cable Cross-Sections

- Optical micrographs show, as expected:
  - Uniform strand cross-sections in the middle of the cable width
  - Significant distortion of module geometry and barrier thinning in the most deformed edge location

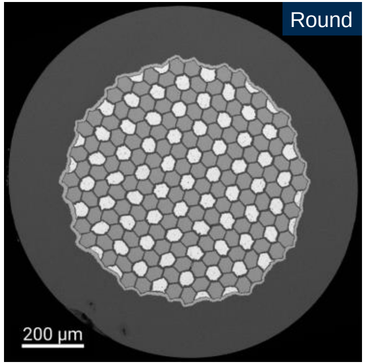



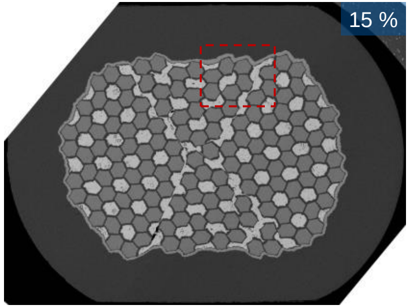



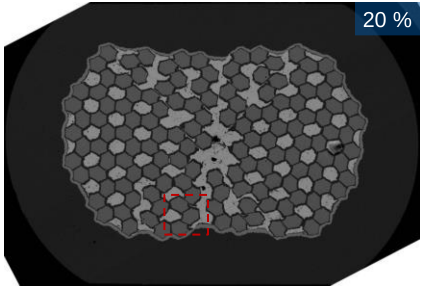


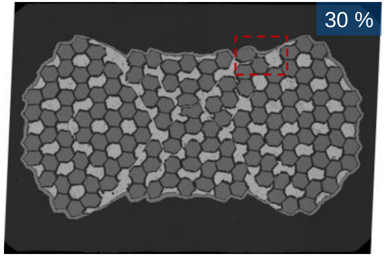
# JASTEC Rolling: Key Features

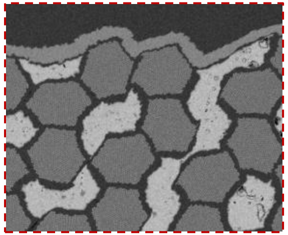
- As observed for previous DT wires, Sn regions deform and merge, whilst Nb modules are largely displaced intact



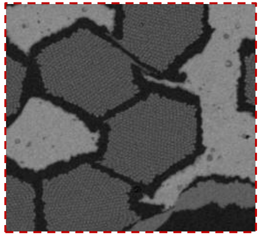



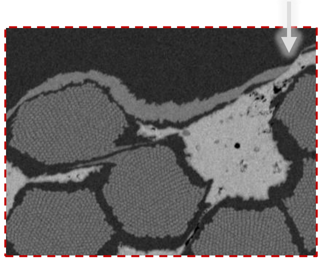



Relative displacement of Nb modules; Sn core merging



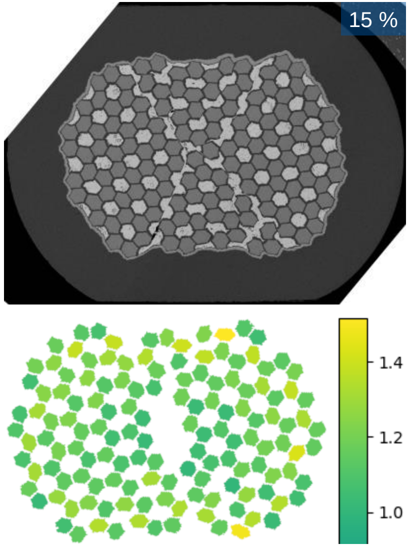
Shearing at periphery of Nb modules; barrier thinning

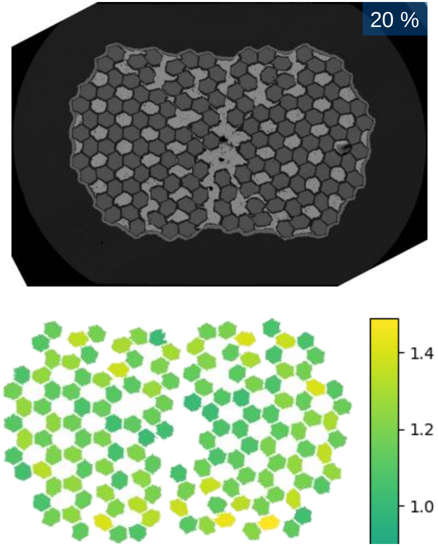


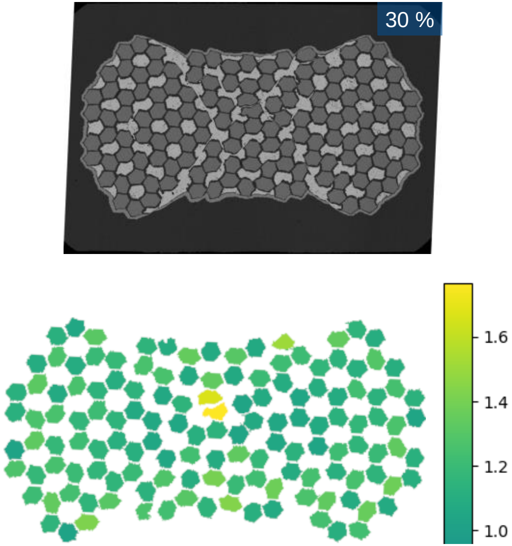
Barrier breakage, extended Nb module shearing

# JASTEC Rolling: Aspect Ratios

- Aspect ratios do not show large increases, or form bands relative to the rolling direction:
  - Unlike RRP® or PIT – see 3-MO-CS2-06S this afternoon
- Large variation in Nb modules between longitudinal positions, as broad Sn regions can open up locally

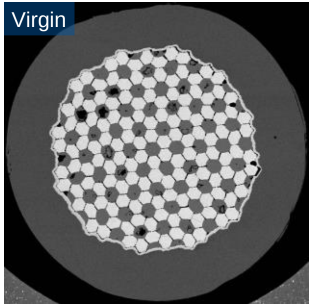


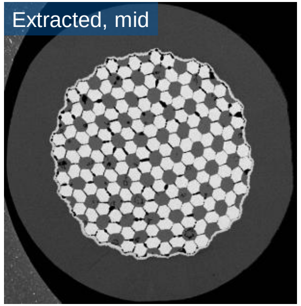



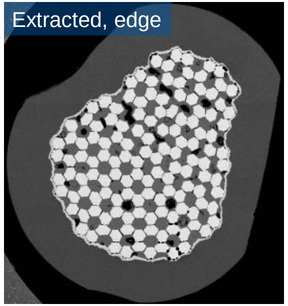



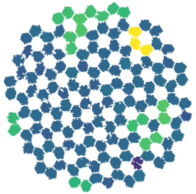
# JASTEC: Reacted Strands

- After heat treatment and the Nb → $Nb_3Sn$ volume expansion, the separation of modules is locally small
  - Difficult to be certain if modules are in contact: colour maps below show **area**, but can be considered to highlight potential clusters of different numbers of modules in contact
  - As expected, the most distorted position at the cable edge exhibits larger clusters of modules potentially in contact → potential increase in $d_{eff}$ and/or stability impact
- Considering only well-separated modules, the distribution of aspect ratios remains comparable to unreacted samples

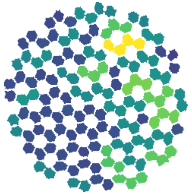


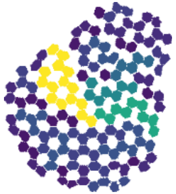



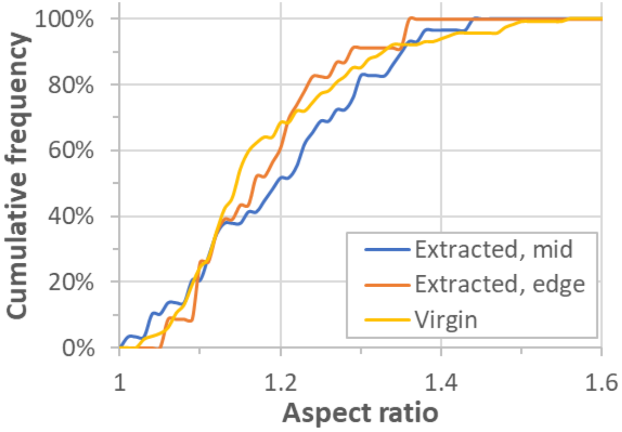



Area distributions



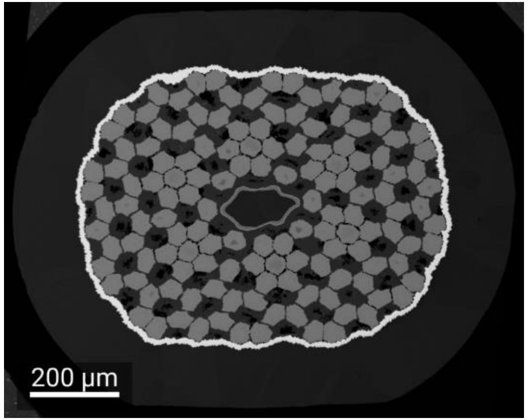


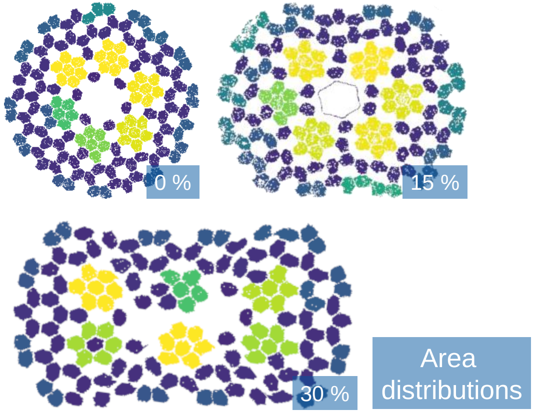


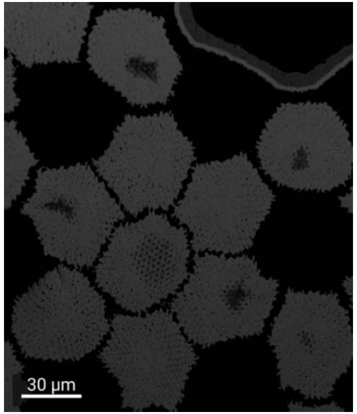

Aspect ratio distribution of well separated modules

# KAT: Reacted Strands

- For KAT reacted strands, observations for subelement shape are similar to JASTEC
- In addition:
  - The area distribution plots highlight 6 clusters of $Nb_3Sn$ regions in close proximity after reaction, which could contribute to increase $d_{eff}$
  - For larger rolling reductions, the internal diffusion barrier is severely deformed
    - The internal Cu volume is small, and not critical to Cu/non-Cu, but it may influence the deformation of neighbouring modules
  - Unreacted Nb is evident in the centre of some Nb modules, indicating the potential for increase $I_c$ if tin supply can be improved by design or heat treatment

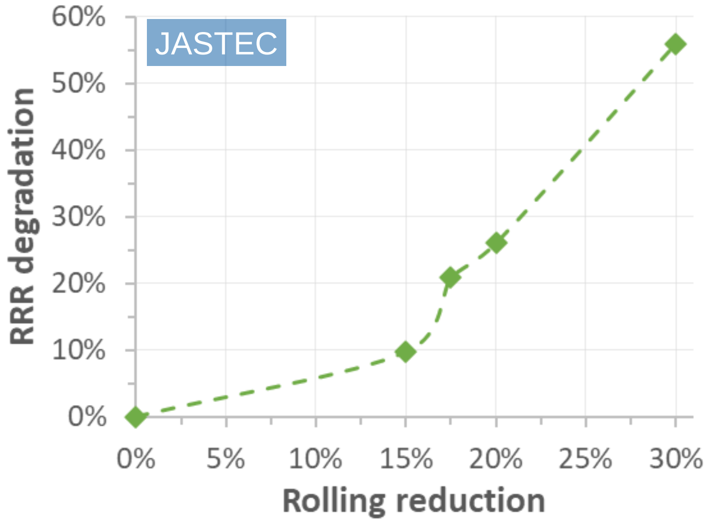


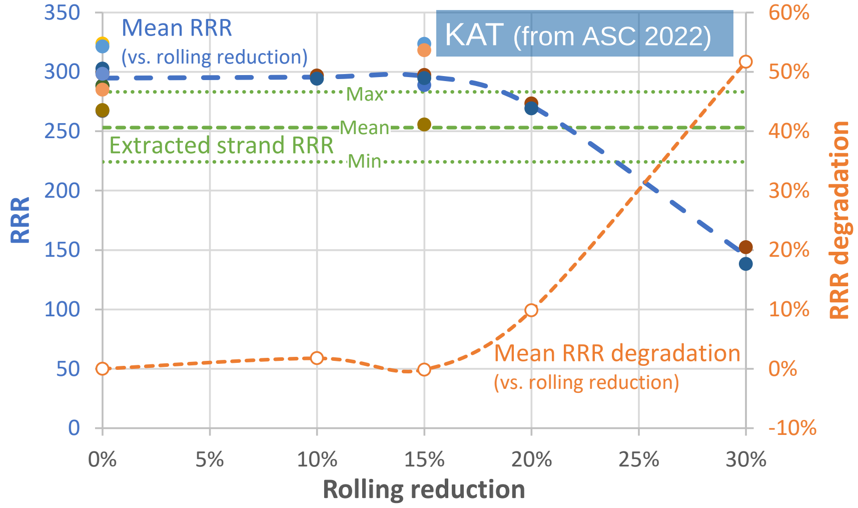



# RRR

- The RRR of JASTEC and KAT rolled samples and extracted strands is extremely high due to the largely intact external diffusion barrier
  - RRR degradation reached ~50 % at 30 % rolling reduction for both JASTEC and KAT wire
  - RRR degradation appears a little higher at small rolling reductions for JASTEC, but few samples tested



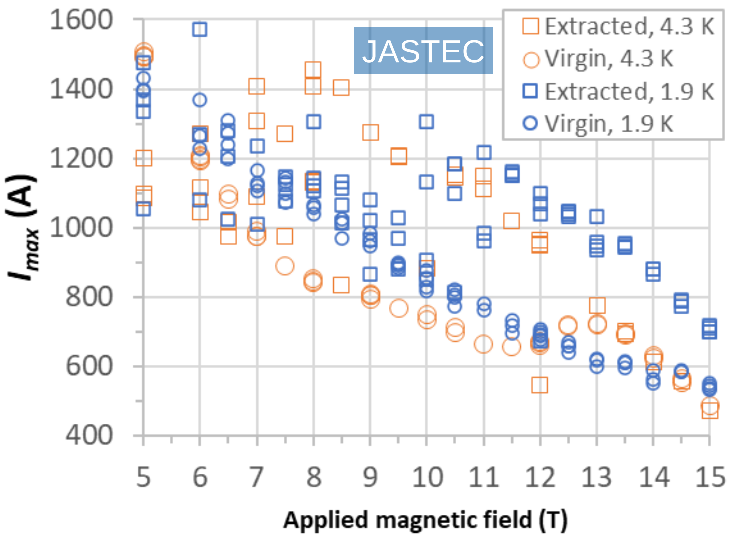



# Critical Current and Stability (1)

- For both the present DT wires, samples have frequently quenched in $I_c$ testing at 12–15 T: difficult to obtain robust statistics for $I_c(B)$ performance
- Self-field instability would be expected to be challenging for these large diameter (1.0 and 1.1 mm), high $J_c$ designs
  - Increased $d_{eff}$ locally, e.g. at contact between subelements, may also contribute to magnetisation instability
- To evaluate the behaviour, multiple $V$–$I$ transport measurements have been performed at both 4.3 K and 1.9 K:
  - Multiple measurements at each test field, starting at 15 T and proceeding downwards in small steps
  - Samples tested in pairs of different deformation state (rolled, extracted)

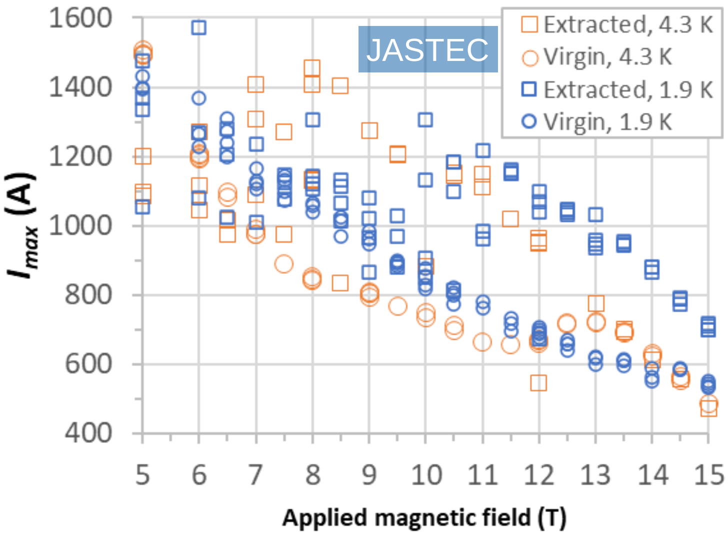
# Critical Current and Stability (2)

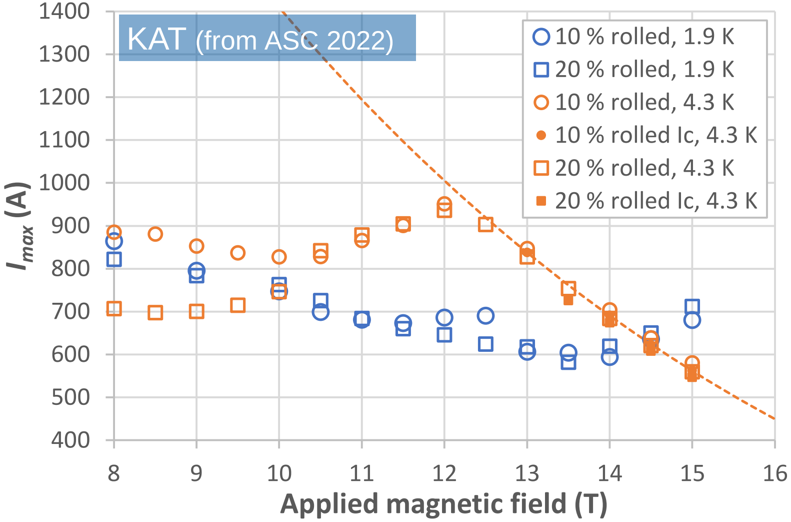
- For the JASTEC wire:
  - A strand **extracted** from the cable appears to quench close to the onset of the $I_c$ transition at:
    - 1.9 K: 12 T and above
    - 4.3 K: ~8 T and above in most cases
  - Extracted strand quench current curves at 1.9 K and 4.3 K intersect at ~ 8 T
  - For the **virgin** (round) wire:
    - At 4.3 K, values appear to be stability limited below ~13.5 T
    - Subsequent quench currents at 4.3 K, and all values at 1.9 K, are anomalously low compared to the extracted strand
      - Additional samples currently in testing



# Critical Current and Stability (3)

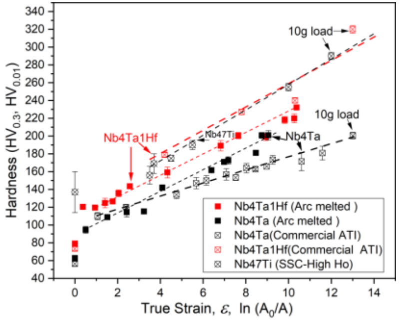
- Comparing the JASTEC extracted strand and KAT samples, performance is less severely stability limited at low field for the JASTEC wire
  - This could potentially be addressed by heat treatment optimisation as well as design changes
  - Further tests are in progress



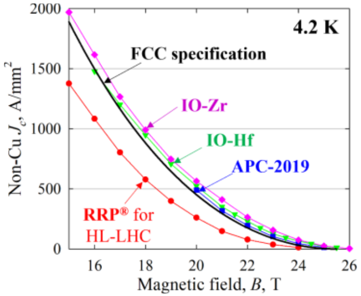



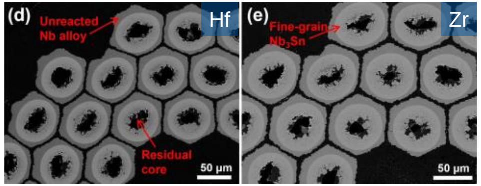
# Future Prospects: Hf, Internal Oxidation

- A significant increase in $J_c$ (relative to the RRP® baseline) probably requires new approaches:
  - Hf alloying was proposed to cause $Nb_3Sn$ grain refinement by suppressing Nb alloy recrystallisation (NHFML, FSU, US)
    - S. Balachandran et al., *Supercond. Sci. Technol.* **32** 044006 (2019)
  - Internal oxidation of Zr or Hf in Nb alloys forms oxide precipitates, acting as pinning centres and refining $Nb_3Sn$ grain sizes
    - X. Xu et al., Appl. Phys. Lett. **104** (8) 082602 (2014)
- Internal oxidation has been implemented in both PIT and internal tin wire types
  - The hardening behaviour of Hf-alloyed Nb-Ta poses some challenges in wire drawing, and potentially also in subsequent cabling
- PIT wires produced at Hyper Tech (in collaboration with Fermilab and OSU) have shown excellent $J_c$, but:
  - There is currently limited validation of stability and cabling behaviour
  - Many of the same optimisation challenges may apply as for conventional Bruker PIT wires
- Rod-in-tube wires are under development at UNIGE in collaboration with CERN
  - A similar $J_c$ enhancement has been observed in model samples, and wire development in progress
  - An oxide powder is needed as an oxygen source: its configuration will also affect drawing and cabling behaviour



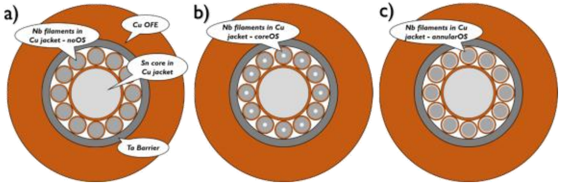

S. Balachandran et al., https://dx.doi.org/10.2139/ssrn.4303410




SEM cross-sections of reacted Hyper Tech Hf- and Zr- alloyed internal oxidation PIT wires

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




Candidate oxygen source configurations for rod-in-tube wires

G. Bovone et al., *Supercond. Sci. Technol.* **36** 095018 (2023)

# Summary (1)

- The different deformation characteristics of RRP®, PIT (BB) and DT wires have been assessed by electron microscopy, image analysis, and $I_c$ and RRR measurement
  - RRP® and PIT wires show a similar evolution of subelement/filament aspect ratio, but the latter is more susceptible to $I_c$ degradation
  - For DT wires, the deformation is mostly accommodated by displacement of Nb modules and deformation of Sn modules
  - $I_c$ degradation is significantly higher (but still acceptably low, <5 %) for RRP® 162/169 (1.1 mm) than 108/127 (0.85 mm)
- 15 % rolled samples underestimate cabling degradation of $I_c$ (17.5 % rolling may be more representative), but conservatively overestimate RRR degradation (as severe degradation is localised to the edges)
  - Work is in progress to evaluate RRR locally at the cable edge
  - Modelling of the strand deformation during rolling and cabling is planned

# Summary (2)

- Distributed tin wires with comparable subelement size produced by JASTEC and KAT have been successfully cabled
- Deformation behaviour is broadly similar for both: Nb modules tend to move before they deform, with little increase in aspect ratio
  - RRR degradation on rolling and cabling is low, from a high baseline
  - There is little indication of $I_c$ degradation on rolling or cabling, but data are limited due to stability issues
- For JASTEC strand extracted from a cable, quench currents at 1.9 K appear close to $I_c$, but results for the virgin strand were inconsistent
  - Tests of additional samples are in progress
- There are promising indications for the potential of distributed tin wires, and that further heat treatment optimisation would be beneficial
- Future trends in wire development, and the sensitivity of deformation behaviour to exact wire design and heat treatment, demonstrate the need for further study.

# Acknowledgements

- The efforts of all our wire development collaboration partners in the HFM programme are gratefully acknowledged, and in particular:
  - For distributed tin wires: KEK, JASTEC, KAT
  - For internal oxidation R&D: UNIGE
- Warm thanks to all members of TE-MSC-LSC involved in cabling, sample preparation and testing of the wires in buildings 103 and 163