



Advanced Examination of Nb₃Sn Coils and Conductors for the LHC luminosity upgrade: Computed Tomography and Materialographic Analyses

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Outline

- 1. Investigation scope
- 2. Method and techniques
- 3. Deployment on 11T dipole coils, Computed Tomography (CT) and Materialographic Analyses
- 4. MQXF quadrupole coils, local investigations and destructive examinations
 - a. CERN MQXFB coils, extended inspections by deep Copper etching and subsidiary NDT techniques
 - Tested coils (CR108)
 - Virgin coils (CR120, CR126)
 - b. Confirmation of the method and investigation of physical quenches on US MQXFA coils:
 - Coil end and wedge/end spacer transitions (AUP 214, AUP 213 from non-conforming magnets)
- 5. Considerations and conclusions



1. Investigation scope

Possible causes of degradation:

- Issues internal to the coil Shear and bending loads on unsupported wire
 - Strand / wire is locally supported by the resin system
 - Presence of strands dislocation (pop-in/pop-out)?
- Occurrence of cracks in filaments
 - Extent and severity of possible flaws
- o Insulation imperfections, lack of bonding, shrinkage cavities, cracks



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Front view

1:7

4

magnets 1

2. Method and techniques

Experimental procedure

- Global, High Energy Linac X-ray Computed Tomography (CT) to identify possible events in a NDT manner in the whole volume at the mesoscopic level
- 2. Identification of Volumes Of Interest (VOI)

F. Lackner et al., IEEE Trans. Applied

Superconductivity, vol. 28 (2018) 1-5

- 3. Definition of a removal plan and cutting route
- 4. Localized, High Resolution in-house X-ray Computed Tomography of selected VOI for detail localization
- 5. Microscopical analysis and destructive tests (DT)

Samples issued from the 4.2 m long AUP 213 coil of the MQXFA08 magnet \Downarrow

↑ Short model 11 T dipole coil C122 (coil)

head), connection side



MQXFB CR108, 7.281 m long – in 2017 longest

Nb₃Sn coils fabricated so far for accelerator



2. Method and techniques, NDT

\Downarrow 6 MeV Linac CT - TEC-Eurolab Modena /IT on a Diondo device, then Diondo GmbH /DE

Resolution: 120 µm Spot size: 2 mm Energy: 6 MeV



5.900 (mm)

3/6/9[MeV]

X-Ray Source

Detector

Scan Volume, maximum Focus-Detector-Distance Sample Weight System Dimensions System Weight Manipulation Flat Panel Detector 3.000 x 3.000 px, 140 [µm] Ø 700 x 1000 H [mm] 4000 [mm] 200 [kg] L 5.900 x B 1.500 x H 2.900 [mm] 17 [t] granite based, 6 / 7 axes,

Microfocus X-ray tube: Max. voltage 225 kV Max. current 3000 μA Max power 500 W

- Min. focal spot size **7 μm**
- High resolution flat panel: 40 x 40 cm
 2048 x 2048 pixels, 16 bit
 - 2048 x 2048 pixels, 16 bit
- Tube-detector distance: 1375 mm
- Max. spatial resolution: 4 μm



$\widehat{\uparrow}$ In house Zeiss Metrotom 1500 CT acquired in 2017 \Downarrow



Advanced Examina

2. Method and techniques, DT



Advanced material investigation facilities, see <u>EN-MME-MM</u> \Rightarrow :

- sample preparation (grinding and polishing)
- microscopy observation (optical microscopy, advanced high resolution electron microscopy including FIB, local strain mapping using EBSD technique, quantification of crack distribution)
- Micro- (soon nano-) hardness, mechanical testing



Preparation



Microoptical observation

↑ Diamond WireTec /DE saw DW.350-L for high precision cutting (DWS) Deep copper etching based on ASTM E340 50% HNO₃ macroetching for Cu and brasses

On polished or as cut and ground surfaces







Advanced electron microscopy including FIB-SEM and a wide range of detectors (EDS, EBSD, low voltage EDS)

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3. Deployment on 11T dipole coils, CT and Materialographic Analyses

Magnet P1 – Hybrid assembly

Collared Coils: Aperture D1: CC01, Coils: GE02_Upper and GE03_Lower **dismounted in May 2019**

coil GE02 (limiting coil of 1st hybrid assembly)

- coil02 NCS (GE02)
- coil02 CS (GEC02)

Suspected causes of degradation:

- Locations of concern, like between inner / outer layer, and at the interface between the turns and the head spacers
- Most probably origin of the quench in the inner layer close to the coil pole







3. Deployment on 11T dipole coils, CT and **Materialographic Analyses**

Coil GEC02

Coil head connection side **Received in September 2020**



Coil GE02

Coil head non-connection side Likely with damages – Quenches Supposedly w/o damages – No quench, Received in July 2020 for NDT feasibility test



Coil GE11 Coil head connection side virgin state **Received** in February 2021





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3. 11T, CT Analyses

CERN

Coil GEC02 – CT: quenches localized on this side



3. 11T, CT Analyses Coil GE02 – CT: No quenches localized on this side, no significant events



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3. 11T, CT Analyses

Coil GE02 – CT: No quenches localized on this side, no significant events



Event 1 – first end cable in inner layer, Misaligned strands (pop-in / pop-out)

3. 11T, CT Analyses

Coil GEC02 – **Events**



Event 2 – vicinity of fourth spacer in inner layer,

Bulged cable





10000µm

3. 11T, CT Analyses

Coil GEC02 – Event 1





3. 11T, CT Analyses





3. 11T, (& MQXF) CT Analyses - Comparison of coil end status (bulged and popped in/out strands)



AUP P06 NCS, internal layer MQXF 107 NCS, internal layer



MQXF 108 NCS, internal layer

3. 11T, (& MQXF) CT Analyses - Comparison of coil end status (bulged and popped in/out strands)

AUP 214 NCS, internal layer AUP 108 NCS, internal layer



Linac CT of coil heads:

- Allows identifying VOI affected by bulging and popped in/out strand events
- Thorough comparison of the outcome of winding
- Indispensable prior to metallographic cuts at coil heads

3. Deployment on 11T, Materialographic Analyses







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3. 11T, Materialographic Analyses





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Metallography GEC02 – Event 1

Extremity straight cut, mirror to the detached portion, reveals in as-cut state:

- Lack of bonding between cable of interest, spacers and insulation is highlighted at the mirror cut in correspondence of the detached portion,
- Environment of the cable of interest is preserved on this cut and has been studied

Polished surface of the present cut allowed to show that:

- Pop-out/pop-in strands are embedded in resin
- SC appear free from cracks in this position
- Cracks in insulation and porosities can be observed, especially at the vicinity of spacers
- Copper composing the strands is at annealed state (approx. 55-60 HV0.1)









20 µm

Metallography GEC02 – Event 1

Τί Κα1

Cable of interest detailed examination shows:

- The presence of angular and radial cracks at indicated plane (few SC impacted from pop-out and other strands of the cable)
- SEM-EDS confirmed that phases in presence in sub-elements composing the SC are the one expected after reaction
- Pop-out/pop-in strands are embedded in resin (some porosities are observed)
- Copper composing the strands is in annealed state (approx. 55-60 HV0.1)



Filaments (belonging to different strands) are affected by cracks in the SC phase





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3. 11T, Extension in axial direction of radial cracks, assessed by FIB-SEM (C122 coil)





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2022.10.24

l Probe =

I Probe = 500 pA WD = 5.1 mm Mag = 20.00 K X Adrienn Baris EHT = 3.00 kV Detector = InLens 29 Sep 2021



3. 11T, FIB-SEM (C122 coil)



Morphology of crack 1, 22.7 µm deep

Crack 2, phases analysed by EDS, presence of Nb, Sn and traces of Cu





Nb Lα1 Map Data 1 -...



2.5µm



2.5µm



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4. MQXF quadrupole coils, local investigations and destructive examinations - MQXFB CR108



Franco J. Mangiarotti

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4. MQXFB CR108, CT

CT of straight part segment CR108LJ and CR108OLJ





- Absence of bulging and popped in/out strands in straight sections
- Precise dimensional position of references (holes in the Ti-alloy pole) before cut (dimensional metrology tool)





tor 6: 23685 at (-37.26 / 5.30 199.55 mm

24632 at (33.83 / 5.3 / -0.01 mm)

37.97 / 5.30 -202.01 mm

)1.73 mm



4. MQXFB CR108, CT

CT of straight part segment CR108LJ and CR108OLJ



Outcome of CT:

- Absence of bulging and popped in/out strands in straight sections
- Precise dimensional position of references (holes in the Ti-alloy pole) before cut (dimensional metrology tool)
- For the first time, identification through CT of severe cracking in the resin reach areas of Ti alloy pole shims, eventually confirmed by metallography



4. MQXFB CR108, DT

Metallographic examination – planes of interest



4. MQXFB CR108, DT – cross sectional cuts

Example of Cut 4: a thorough examination revealed the presence of micro cracks at SC phase (14 sub-elements for sector A, 85 for sector B and 47 for sector D) that seem to be mainly located in a cloud (stress field?). Only few events (<5) with random location have been identified on C.

Conductor exhibiting micro-cracks (several sub-elements can be impacted for an indicated location)





MQXFB CR108, DT – deep copper etching

Protocol

Planes previously polished and examined are progressively attacked with HNO₃ diluted at 50% in water (ASTM E340 macroetching) with optical examination between each step. Approx. 400 µm to 600 µm of copper are etched out by this mean.



General view of the transverse cross section after etching, example of cut 4



General view of an etched strand, example of cut 4



4. MQXFB CR108, DT – deep copper etching

Several thorough examinations of the whole transverse cross sections between copper dissolution steps and after the last step revealed for cuts #1, #2 and #4 that:

The same and the only one strand at the top edge of the Rutherford cable adjacent to the pole block at inner layer exhibits collapsed SC filaments. The portion of the affected strand where filaments are broken is consistent in the three cuts. Cut 3b does not exhibit broken filaments.





4. MQXFB CR108, DT – deep copper etching , SEM examination

SEM examination allows collapsed filaments and fractured SC sub-elements to be to highlighted. Some filaments are missing, they probably withdrew in rinsing water after etching.





4. MQXFB CR108, DT – deep copper etching , SEM examination

Some filaments are missing (probably withdrew in rinsing water after etching); filaments that seem still intact: 75/108, 76/108, 74/108 for cut 1, 2, 4, respectively.





4. MQXFB CR108, DT – deep copper etching , SEM examination

Highlight on fractured filament exhibiting transverse plane fracture: the fractured surface appears perpendicular to filament axis with a typical aspect of a bri





4. MQXFB CR108, DT – longitudinal cuts

Extraction of the VOI

Extraction of the VOI containing the cables at pole block inner layer has been carried out by DWS.





4. MQXFB CR108, DT – longitudinal cuts

etching

Bottom subjected to acid

Optical examination

Progressive polishing with intermediate observation steps has been performed to access to 1st and 2nd row's midplane of the first Rutherford cable. The 1st row's midplane exhibits events at superconductive filaments.





2nd row's midplane

4. MQXFB CR108, longitudinal cuts – quench location A (LJ) Optical examination – 1st row's midplane



Examination of the whole surface reveals large cracks with clearly cracked filaments in several strands at the top edge of the Rutherford cable close to titanium poles.

> Opened and cracked filaments seem to exhibit a V shape which could indicate bending issue.



4. MQXFB CR108, extended inspection by deep copper etching



- Reminder of further steps envisaged for coil CR108
- Assessment of large volumes through DWS cutting followed by light grinding and deep Copper etching
- **2 x 1 m coil lengths** to be explored symmetrically around A & B quench locations
- 2 x 20 slices x (20 h cutting + 4 h etching and examination) = 320 h (40 d / 8 w) + 560 h as background task
- tbc based on first examined samples

Analysis type	Performed by	Cutting Date	Analysis Date	Longitudinal position (Distance from LE end, mm)	Results
omography&Metallography	EN/MME/MM	Oct-20	Dec-21	150-450	CERN-0000218257
Image Analysis	TE/MSC/LMF	Oct-20	Feb-21	450-500	EDMS 2479219
Mechanical	FNAL	Apr-21	Sep-21	640-740	
Mechanical	FNAL	Apr-21	Sep-21	740-840	
Heat Transfer	TE/CRG/CI	Apr-21	Nov-21	840-975	Indico 1147897
Metallography	EN/MME/MM	Oct-20	Jun-21	3460-3860 LJ/OLJ	EDMS 2646593
Tomography	EN/MME/MM	Oct-20	Jan-21	3460-3860 LJ/OLJ	EDMS 2605729
Metallography	TE/MSC/SCD	Nov-21	ongoing	4750-4800	
Metallography	TE/MSC/SCD	Nov-21	ongoing	4800-4850	
Metallography	EN/MME/MM	Mar-22	Apr-22	6540-6580	indico 1145267
Metallography	EN/MME/MM	Mar-22	Apr-22	6580-6620	Indico 1145267
Mechanical	FNAL	Apr-21	Sep-21	6781-6881	
Mechanical	FNAL	Apr-21	Sep-21	6881-6981	
omography&Metallography	EN/MME/MM	Oct-20	Dec-21	7181-7481	EDMS 2605746

<u>Courtesy S. Izquierdo, further details, see:</u> <u>https://indico.cern.ch/event/1145267/</u>



4. Extended inspection by deep copper etching: MQXFB CR108, EDMS_2731361

Sketches not at scale **Results – 3750-4750** \downarrow Front view 4700 3750 No events observed Three positions 300 mm, 650 mm and 700 mm from quench region exhibit damaged strand Strand(s) with collapsed filaments





4. Extended inspection by deep copper etching: MQXFB CR108, EDMS_2731361



Observed events are in correspondence to titanium pole ends



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4. Extended inspection by deep copper etching: MQXFB CR108, EDMS_2731361





4. Extended inspection by deep copper etching: MQXFB CR108, longitudinal cuts, EDMS_2731361

- Longitudinal cuts of the 108 coil-samples 2670 and 3900 (both presenting crack-events as previously shown)
- The longitudinal cut 2670 was found to have approx. 240 crack-events spread over the longitudinal crosssection, especially close to the region where Cu was previously etched away.





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4.b MQXFA07 - AUP 214 CS metallurgical inspection, EDMS 2739504

VTAD TLIRN A

Coil AUP214 from magnet MQXFA07 after initial ramp-up was limited by several quenches below nominal current during training. According to voltage tabs, the portion involved into limiting the performance is located in between turn 2 to turn 6, inner layer, in coil's head at connection side.

Prior NDT on both coil's heads revealed the presence of bulged cables and some popped out strands.

Transverse cross sections covering transitions end spacer and wedge – 1st





A01

A05

A06

4.b MQXFA07 - AUP 214 CS metallurgical inspection, EDMS_2739504

1st priority cut - Transition between end spacer and wedge

10 mm thick slice covering the transition between end spacer and wedge.

Two halves have been ground and polished to approach the interface.





Advanced Examination of Nb₃Sn Coils and Conductors for the LHC luminosity...

4.b MQXFA07 - AUP 214 CS metallurgical inspection, EDMS_2739504

1st priority cut - Transition between end spacer and wedge

LJ & OLJ side – deep copper etching



Copper dissolution with 50% HNO3 (according to ASTM E340).

450 µm-600 µm depth has been dissolved

Not a single strand exhibits collapsed filaments.

The same result was found for the OLJ side.





4.b MQXFA07 - AUP 214 CS met

1st priority cut - Transition between end s

Few cracks at midplane block in a delimited area A localized field of transverse cracks at SC filaments is observed at pole block

Original high-definition stitched picture are set out in annexes





5000 μm

Imber 5000 µm

4.b MQXFA07 - AUP 214 CS metallurgical inspection, EDMS 27

1st priority cut - Transition between end spacer and wedge

OLJ side – longitudinal cut 2nd row



No transverse cracks at SC filaments are observed at pole block 2nd row of Rutherford cable





4.b MQXFA07 - AUP 214 CS metallurgical inspection, EDMS_2739504

1st priority cut - Transition between end spacer and wedge





n, <u>EDMS_2739504</u>



Height = 857.2 µm

WD = 4.7 mm

Signal A = SE2

Aperture Size = 60.00 µm

Date: 28 Jun 2022

5. Considerations and conclusions

- A sequence of NDT examinations through large volume Linac and local CT of VOI followed by cross-sectional, deep Copper etching and longitudinal observations of cracked filaments allowed to univocally identify physical events associated to the quenches (broken filaments in strands at specific positions) – previously identified using other tests (e.g. voltage tapes for CR108 coil)
- The longitudinal extent of the damage of the affected Rutherford cables can be assessed through longitudinal cuts following deep Copper etching
- Extended and/or severe conductor damage is almost systematically identified in correspondence of transitions or singularities:
 - Vicinity of spacers (bulging and popped in/out strand events)
 - Titanium pole ends (broken filaments)
 - End spacers wedge transitions (large field of cracks)
- Assessment of the resin status is straightforward in cross-sectional cuts
- The applied sequence of NDT + DT examinations is reconfirmed as a powerful tool to identify conductor imperfections and/or damage of the superconducting phase, and to assess the severity of such damage





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3. 11T, (& MQXF) CT Analyses - Comparison of coil end status (bulged and popped in/out strands)

*outer layer of coil MQXFB 120 is not representative (absence of binder)

Name	State	Popped in/out strands events Outer Layer	Popped in/out strands events Inner Layer	EDMS report	
MQXFS 106 CS	Limiting MQXFS3c	14	6	<u>2667969</u>	
MQXFS 106 NCS	Limiting MQXFS3c	13	5		
MQXFS 107 CS	Reference CERN	2	1	<u>2646366</u>	
MQXFS 107 NCS	Reference CERN	0	1	<u>2605746</u>	
MQXFB 108 CS	Limiting coil	9	10	<u>2667966</u>	
MQXFB 108 NCS	Limiting coil	13	11	<u>2605746</u>	
MQXFB 108 Straight	Limiting coil	0	0	<u>2605729</u> <u>2605732</u>	
MQXFB 120 CS	Winding inner layer	(15)*	1	<u>2668915</u>	
MQXFB 120 NCS	Winding inner layer	(11)*	1		
AUP_P06 NCS	Reference USA	0	0	<u>2646366</u>	
AUP_108 CS	USA- SELVA winding	2	0	<u>2683078</u>	
AUP_108 NCS	USA- SELVA winding	5	2		
AUP_214 CS	USA – Limiting coil	3	2	<u>2721396</u>	
AUP_214 NCS	USA – Limiting coil	5	2		



4.b MQXFA07 - AUP 214 CS metallurgical inspection, EDMS_2739504

1st priority cut - Transition between end spacer and wedge

LJ side



Some "usual" shrinkage cavities through glass fibres/resin matrix are identified. Ceramic coating on end spacer ensures good cohesion with resin.

Inspection of SC filaments did not reveal any major events. Some radial "closed" microcracks can be observed.

For the OLJ side, very similar results were found (here also some cracks through the glass fibres/resin matrix were identified)





4. Extended inspection by deep copper etching of longitudinal cuts 2670 and 3900 : MQXFB CR108, <u>EDMS_2731361</u>

The major events were found at the top part of strand 1 up to strand 7. For strand 3 and 6, the "V" shape was identified.



Hole corresponding to the longitudinal cut 2670

In this sample, only the pole-to-pole transition at the inner layer was present.



For the longitudinal cut 3900, an extent of the events of approx. 15 mm was observed at the top part (from strand 1 to strand 6). For this specimen, only few minor events (15-20) were observed over the cross-section, in the region close to the etched side.



4. MQXFB CR108, DT – cracks and stress field

Courtesy S. Izquierdo Bermudez, https://indico.cern.ch/event/1150724/ and this conference 1LOr2A-03

Radial cracks and stress map

The higher concentration of micro-cracks in the analyzed sections of coil 108 straight section is in IL the mid- plane block (see talk from S. Sgobba)

Coil 108, coil straight section



Remark: In the case of BP1, with a maximum coil stress of 100 MPa at warm after loading, we expect 135 MPa in the pole block after cool down and a maximum of 110 MPa in the mid-plane at 15 kA



(120 MPa at I_{ult})

