European Nb_3Sn Superconducting Strand Production and Characterization for ITER TF Coil Conductor

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***Abstract*—The European Union contributes around 20% of the cable-in-conduit conductor lengths needed for the ITER toroidal field (TF) magnet coils. For that purpose, 97 tons of Nb_3Sn superconducting strand have been fabricated over five years, the production being completed in 2014. This superconducting strand has been manufactured by two companies, namely, Bruker EAS (Germany) and OST (USA), through the bronze route and the internal tin diffusion, respectively. This paper reports the outcomes of this strand mass production and of the strand characterization as performed by the suppliers and cross-checked on a regular basis by Durham University.**

***Index Terms*—Cable-in-conduit-conductor, fusion magnets, low-temperature superconductors, superconducting wires.**

I. INTRODUCTION

THE main objective of the ITER project is to construct a fusion reactor in order to confirm the technological feasibility of fusion power. ITER, which is an international collaboration including China, the European Union, India, Japan, the Russian Federation, South Korea and the USA, is being built close to Saint-Paul-lez-Durance in the south of France[1].

The European Joint Undertaking for ITER and the Development of Fusion Energy ("Fusion for Energy" or F4E) supervises the European contribution to ITER [1], [2]. Among others, F4E is in charge of the procurement of 20 km of Cable-In-Conduit Conductor for Toroidal Field (TF) coils [3]. This amount represents around 20% of total amount of conductor needed for all ITER TF coils. The TF conductors are constituted of copper and Nb_3Sn strands first cabled and then inserted into a stainless steel jacket [4].

For the manufacturing of the European TF coil conductors, 97 tons of Nb_3Sn strand have been fabricated over five years by two companies: Bruker EAS GmbH (Germany) and Oxford Instruments Superconducting Technology (New-Jersey, USA).

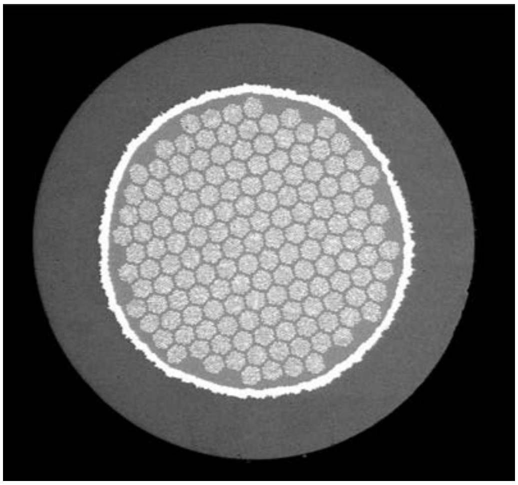

Fig. 1. Cross section of the BEAS Nb_3Sn strand. It contains 151 preconductors surrounded by a tantalum diffusion barrier.

In this paper, the main outcomes of the mass Nb_3Sn strand production and of the strand characterization as performed by the suppliers and cross-checked on a regular basis by Durham University are presented.

II. NB_3SN STRAND PRODUCTION

A. Bruker EAS

The contract between Bruker EAS GmbH (BEAS) and F4E was signed in December 2009 for the supply of 37.7 tons of chromium-plated Nb_3Sn strand.

BEAS strand is a quaternary Nb_3Sn wire (both Ti and Ta doping) fabricated by the Bronze Route Process, using a double stacking and extrusion process. As a first step, a preconductor is fabricated by inserting 55 NbTa rods into a CuSnTi matrix followed by extrusion and drawing of the obtained stack. In a second step, 151 hexagonally-shaped pre-conductors are assembled into an Oxygen-Free Copper tube with a tantalum barrier between the pre-conductors and the tube. This final assembly (billet) is then extruded and drawn down to the final strand diameter (0.82 mm). Afterwards the strand is electrolytically coated with a chromium layer of 1–2 μm. A BEAS billet may yield up to 23 km of strand. A cross-section of the final strand is given in Fig. 1.

The ramp-up phase to prepare the facilities and procedures for the strand mass production including the optimization of the Cr-coating parameters took around a year. During the mass production the monthly production rate was around 0.9 ton

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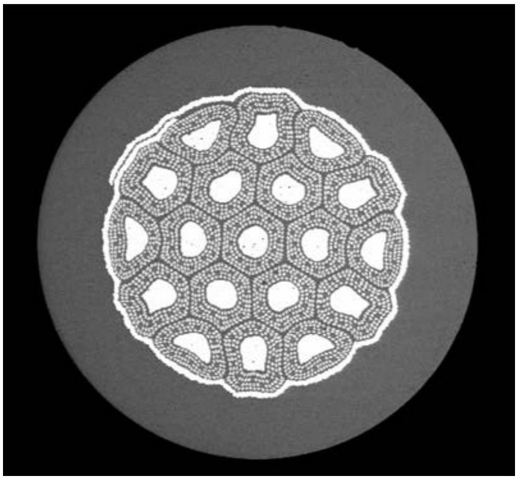

Fig. 2. Cross-sectional view of an OST strand for the TF conductor. It includes 19 sub-elements and a Ta diffusion barrier.

of strand. The strand production was completed in July 2014. Over the full production, an average of 2.8 piece lengths with a length of 7600 m has been obtained per billet. In addition more than 90% of the billets exhibited a yield larger than 80% thus confirming the good workability of the process.

B. OST

The contract between Oxford Instruments Superconducting Technology (OST) and Fusion for Energy was signed in August 2009 for the supply of 59.7 tons of chromium-coated Nb$_3$Sn strand.

OST strand is a ternary Nb$_3$Sn wire (doped with titanium) manufactured by means of the Internal Tin Diffusion Process. First, the sub-element billet is produced by inserting 156 Nb and NbTi rods into an Oxygen-Free Copper billet. After extruding the assembled billet, the billet is drilled in its center and a tin rod is inserted. Subsequently the sub-element is drawn. In a second stage, 19 sub-element rods are assembled together with a tantalum sheet as a diffusion barrier into an Oxygen-Free Copper tube to constitute the final billet. This billet is then drawn down to the final strand diameter. The obtained strand is afterwards plated with a chromium layer. An OST billet may yield a maximum strand length of 13 km. A cross-section view of the OST strand is given in Fig. 2.

In the case of OST production, about a year was also required to adapt the company premises to the strand mass production. During the mass production the strand fabrication rate was about 1.4 tons per month. The strand production has been completed in January 2014. For the entire production, 2.3 strand piece lengths have been obtained per billet as an average. Moreover around 90% of the billets showed a yield larger than 80% demonstrating the fine workability of the manufacturing process.

III. Nb$_3$Sn Strand Characterization

In order to check whether the main strand parameters do comply with the stringent ITER specifications [5], an extensive program of characterization has been set up. Both suppliers performed on every billet acceptance tests either at room temperature (strand diameter, twist pitch, Cr-plating thickness and copper-to-non-copper volume ratio) or at ∼4 K (critical current, Residual Resistivity Ratio (RRR) and hysteresis loss per strand volume over a ± 3 T cycle). More than 8000 low-temperature tests, including critical current, RRR and hysteresis loss measurements, have been performed by both suppliers along the production. In addition, the supplier test data were cross-checked on a regular basis by verification tests performed at Durham University with the collaboration of the University of Twente. At the beginning of the production, 100% of the supplier tests were verified. Afterwards, the sampling quickly reduced to 50% and even 25% of the supplier tests. Altogether more than 4000 low temperature (critical current, RRR and loss) verification tests have been performed. More details on testing procedures can be found in [6], [7].

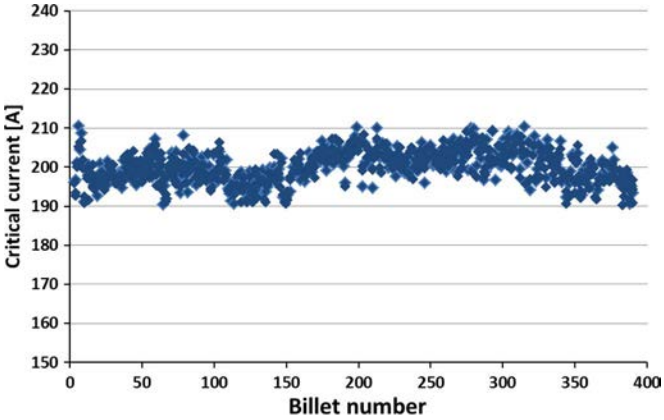

Fig. 3. Critical current at 4.22 K and 12 T of all the strand samples tested at BEAS during the full strand fabrication (specification: >190 A).

A. BEAS Strand Characterization

For the Nb$_3$Sn strand reaction before the characterization of the strand samples BEAS used an optimized heat treatment (HT) schedule with two long duration steps: 160 hours at 595 °C and 320 hours at 620 °C. Thanks to this reaction schedule, BEAS strand typically provides a critical current of 200 A at 4.22 K and under 12 T applied magnetic field as compared to a minimum specified value of 190 A. Fig. 3 shows all the supplier critical current data measured at 4.22 K and 12 T along the full strand fabrication. As shown in Fig. 3, all the critical current values are within specification. Moreover it is noteworthy that the critical current values of all the hundreds of samples tested at BEAS are consistent within ± 5%.

For a given billet, the low variability of critical current data is even more remarkable. Fig. 4 presents the spread in billets of the critical current data measured at BEAS for all the billets for which at least two measurements (point and tail of the billets) have been performed. The scatter, defined as the relative difference between the maximum and minimum values for a given billet, is generally below 2% (or ± 1%) and in a few individual cases about 4%. When considering that this scatter integrates a few factors as testing setup reproducibility, sample mounting and variations in copper-to-non-copper ratio along the billet, one may appreciate the remarkable consistency of the electrical properties of the strand along its length. The

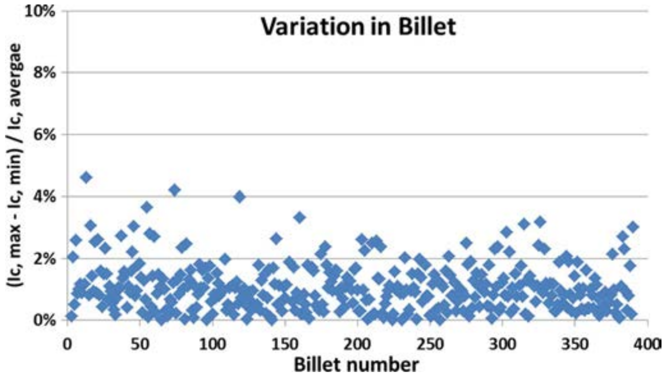

Fig. 4. Variation of critical current values in billets as measured by BEAS at 4.22 K and 12 T and as a function of the billet reference.

TABLE I
MEAN VALUES FOR THE MAIN STRAND PARAMETERS OF BEAS

Parameter	BEAS mean	Durham mean	Specification
Critical current [A]	200	197	>190
n-value	44	43	>20
RRR	117	106	>100
Loss [mJ/cc]	65	58	<500
Cu/non-Cu	0.94	0.93	0.9-1.0
Cr-thickness [µm]	1.3	1.3	1.0-2.0

variability of the RRR in a given billet is obviously larger than that of the critical current due to the substantial RRR sensitivity to local copper impurities or barrier defects. For more than 90% of the billets the RRR scatter is within 10% although in very few cases this spread can reach 30%.

The supplier and verification data of the main strand parameters as averaged over several hundreds of measurements representative of the full production and performed at BEAS and Durham respectively are presented in Table I together with the ITER TF strand specification. For the copper-to-non-copper ratio and the chromium plating thickness, supplier and verification mean data are fully consistent. As shown by Table I, the hysteresis loss values are substantially smaller than the maximum specified value (500 mJ/cc). This is obviously due to the very thin filaments of BEAS strand (3 μm in diameter). The critical current as measured at Durham at 4.22 K and 12 T is on average 1.5% below that measured at BEAS. This tiny difference could be ascribed either to a different strand strain following the sample mounting or to a small discrepancy in the absolute calibration of the temperature sensors used in respective reaction ovens.

Nevertheless, in the case of the RRR, there is a clear deviation between supplier and verification data where Durham RRR values are lower on average by around 9% than BEAS data. Cross-checks made on same physical samples measured at both setups demonstrated that BEAS, Durham and University of Twente provided RRR data consistent within 1–2%. Once the consistency of testing setups was confirmed, the impact of the HT environment has been investigated. For that purpose, ten strand samples coming from different billets have been each cut into three specimens. For each strand sample a specimen has been reacted under argon and tested at BEAS, a second one has been reacted in argon and tested at Durham and a third specimen has been heat treated at CERN under vacuum and tested at Twente University. It appeared that the RRR values of BEAS samples were systematically greater than those of Durham, which were systematically larger than those of Twente. On average the BEAS RRR was 114 as compared to 102 for Durham and 93 for Twente. Therefore it seems that the discrepancy observed between supplier and verification RRR values is mainly due to the impact of the environment during HT and the interplay between atmosphere and contaminants.

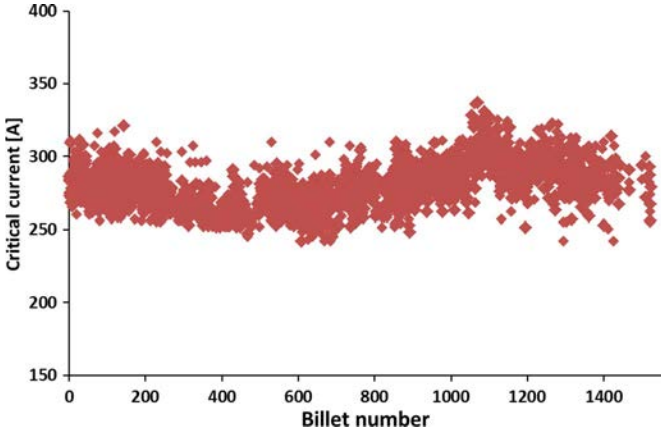

Fig. 5. Critical current at 4.22 K and 12 T of all the strand samples tested at OST during the entire strand fabrication (specification: >190 A).

TABLE II
MEAN VALUES FOR THE MAIN STRAND PARAMETERS OF OST

Parameter	OST mean	Durham mean	Specification
Critical current [A]	281	275	>190
n-value	39	36	>20
RRR	158	143	>100
Loss [mJ/cc]	264	271	<500
Cu/non-Cu	1.00	1.01	0.9-1.0
Cr-thickness [µm]	1.3	1.2	1.0-2.0

B. OST Strand Characterization

For the strand HT prior to characterization of low temperature samples, OST utilized the ITER cycle B, i.e. a plateau of 50 hours at 210 °C, followed by two dwells of 25 hours each at 340 °C and 450 °C respectively and completed by two plateaus of 100 hours each at 575 °C and 650 °C. Once reacted OST strand exhibits a supplier critical current value at 4.22 K and 12 T above 242 A, which represents a minimum margin of 26% as compared to the minimum specified value (190 A) and is quite comfortable. Fig. 5 shows all the critical current values as measured by OST during the entire strand production (more than 2700 samples tested).

The supplier and verification data of the most prominent strand parameters for the complete strand production, as averaged over several thousands of measurements performed at OST and a few hundreds of measurements done at Durham respectively, are listed in Table II.

As shown by Table II, for the hysteresis loss, the copper-to-non-copper ratio and the chromium plating thickness, the values as averaged over OST and Durham measurements are consistent within a few percent. It is worth mentioning that the average loss values are around half of the maximum specified

TABLE III
RRR MEAN VALUES DEPENDING ON REACTION ENVIRONMENT

Strand piece	OST, HT in N_2 (O_2: 2 ppm)	OST, HT in Ar (O_2: 10 ppm)	Durham, HT in vacuum ($\sim 10^{-6}$ mbar)
F0796	144	81	88
F0825	146	131	134

value, the largest value ever measured at OST, 407 mJ/cc, having a comfortable margin of 19% as compared to the specification. Similar to BEAS case the critical current data as measured at Durham are slightly lower than the values measured at OST (by 2% on average).

For the RRR and similar to the case of BEAS, there is an obvious discrepancy between supplier and verification values, where Durham values are lower by about 9% on average when compared to OST data. In order to understand this discrepancy thorough investigations were conducted. First, the testing setups of OST and Durham have been cross-checked by means of same physical samples. Both setups appeared to be consistent within 2%. Considering the HT environment of the RRR samples, OST used nitrogen whereas Durham utilized argon. In order to assess the reaction environment impact on the RRR value, two strand piece lengths of 1000 m each have been cut every 50 m and a strand sample has been taken off. Then the twenty samples for each strand piece have been cut into three specimens. The two first specimens were heat treated at OST oven under nitrogen and argon respectively and afterwards tested by the supplier whereas the third specimen was treated under vacuum at CERN and later tested at Durham. In such a way, the impact of the RRR fluctuations along the piece strand was substantially reduced when comparing the averaged values.

Table III presents the results of the RRR as averaged in the three cases (nitrogen and argon tested at OST and vacuum tested at Durham). As shown by Table III the results are quite puzzling. Indeed if samples reacted in argon provide RRR values comparable to those reacted in vacuum (within 8%) samples reacted in nitrogen have a RRR value systematically higher than those heat treated in argon by 78% on average in the case of F0796 strand piece and 11% for F0825. The reason why two strand pieces with a similar RRR value under nitrogen (144 versus 146) behave so differently under argon (81 versus 131) is far from being clear. Nevertheless it appears that reacting the samples in nitrogen gas apparently enhances the RRR value. In the specific case shown in Table III that is likely due to oxygen residues lower in nitrogen treatment (2 ppm) than in argon reaction (10 ppm). In addition the small oxygen amounts present in the nitrogen gas could create an oxide layer on the strand at the beginning of the reaction [8], [9]. This layer could then act as a diffusion layer protecting the strand copper matrix from further contamination during the HT. Following those investigation results OST changed the oven environment gas to argon for the last third of the production in order to be consistent with ASG, the TF coil manufacturer, which utilizes Ar in the HT of the TF double pancakes.

IV. CONCLUSION

More than 97 tons of superconducting Nb_3Sn strand have been fabricated during around five years for the European ITER TF coil conductors. The production has been completed at mid-2014. During the mass production phase 2.3 tons of strand have been manufactured monthly by BEAS and OST. The strand fabrication by both companies has been characterized by a very good workability demonstrated by a billet yield higher than 80% for most of the billets produced and 2–3 strand pieces achieved per billet on average.

An extensive characterization program has been performed by the supplier in order to ensure the compliance of the strand with the stringent ITER TF strand specification. More than 8000 low temperature acceptance tests have been done altogether by both suppliers. In addition more than 4000 verification tests at low temperature have been done at Durham University and the University of Twente in order to cross-check the supplier tests on a regular basis.

In general a fair agreement between supplier and verification tests has been observed with the exception of the RRR measurements for which verification data are on average 9% below the supplier results. It appeared that this discrepancy is due to the impact of the oven environment during the reaction HT, which should be monitored carefully in particular its oxygen residues.

DISCLAIMER

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