



### **Nb<sub>3</sub>Sn conductor development and characterization for NED**

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### **Abstract**

The main purpose of Next European Dipole (NED) project is to design and to build an Nb<sub>3</sub>Sn ~ 15 T dipole magnet. Due to budget constraints, NED is mainly focused on superconducting cable development and production. In this work, an update is given on the NED conductor development by Alstom-MSA and SMI, which uses, respectively, Internal- Tin-Diffusion and Powder-In-Tube methods, with the aim of reaching a non-copper critical current density of ~ 3000 A/mm<sup>2</sup> at 12 T and 4.2 K. Characterization results, including critical current and magnetization data, are presented and discussed, as well, for conductors already developed by both companies for this project. SMI succeeded to produce a strand with 50 μm diameter filaments and with a critical current of ~ 1400 A at 4.2 K and 12 T, corresponding to a non-copper critical current density of ~ 2500 A/mm<sup>2</sup>. Cabling trials with this strand were successfully carried out at LBNL.

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**Abstract.** The main purpose of Next European Dipole (NED) project is to design and to build an Nb<sub>3</sub>Sn ~ 15 T dipole magnet. Due to budget constraints, NED is mainly focused on superconducting cable development and production. In this work, an update is given on the NED conductor development by Alstom-MSA and SMI, which uses, respectively, Internal-Tin-Diffusion and Powder-In-Tube methods, with the aim of reaching a non-copper critical current density of ~ 3000 A/mm<sup>2</sup> at 12 T and 4.2 K. Characterization results, including critical current and magnetization data, are presented and discussed, as well, for conductors already developed by both companies for this project. SMI succeeded to produce a strand with 50 μm diameter filaments and with a critical current of ~ 1400 A at 4.2 K and 12 T, corresponding to a non-copper critical current density of ~ 2500 A/mm<sup>2</sup>. Cabling trials with this strand were successfully carried out at LBNL.

## 1. Introduction

The Next European Dipole (NED) program is a Joint Research Activity of the Coordinated Accelerator Research in Europe (CARE) project and lies within the framework of EU-FP6 Research Activity [1]. The main initial purpose of NED was designing, developing and building a large aperture (~ 90 mm) and high-field (~ 15 T) accelerator-type dipole magnet made of Nb<sub>3</sub>Sn superconductors. Due to stringent budget constraints, the core of NED program is, for the meantime, the development and the fabrication in collaboration with the European industry of a Rutherford-type Nb<sub>3</sub>Sn cable typically composed of dozens of high-performance superconducting strands. The NED project was launched on January 2004. Following the call for tender in summer 2004, two contracts were signed with Alstom/MSA in France and ShapeMetal Innovation (SMI) in the Netherlands on September

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2004. Alstom/MSA and SMI use, respectively, Internal-Tin-Diffusion (ITD) and Powder-In-Tube (PIT) methods for Nb<sub>3</sub>Sn wire manufacture. The contracts foresee three phases: the first one for the billet design and some cabling tests, the second one for billet design and production methodology refinement and the third one for the fabrication of long length cables.

In this work, after presenting and discussing the NED conductor very demanding specifications, the conductor development carried out so far by both companies will be briefly presented together with relevant characterization results for conductors already fabricated.

## 2. NED conductor specifications and motivations

The ultimate target of NED is to get ready for luminosity upgrade and potential energy upgrade of the Large Hadron Collider (LHC) at CERN [1]. For this purpose, high-field dipoles and high-field gradient quadrupoles will be required. Therefore, there is a genuine need to promote the development of reliable and high-performance Nb<sub>3</sub>Sn wires and cables. Kilometric lengths of such Nb<sub>3</sub>Sn strands should indeed be consistently produced with a high quality in order to fulfill demanding accelerator magnet performances (high critical current density, low magnetization ...).

On the basis of a preliminary magnetic design study [2] and of stability and protection considerations, the specifications of the NED strand were selected [3]. Main strand specifications are summarized in Table 1. Tentative Rutherford cable parameters are presented as well in Table 2.

**Table 1.** Main strand specifications for NED

Strand diameter [mm]	1.250 ± 0.004
Effective filament diameter [μm], design	< 50 μm
Copper-to-non-copper ratio	1.25 ± 0.10
Filament twist pitch [mm]	30 ± 3
Minimal $I_c$ @ 4.22 K and 12 T [A]	1636
Minimal $I_c$ @ 4.22 K and 15 T [A]	818
RRR after full reaction	> 200

**Table 2.** Tentative cable specifications for NED

Number of strands	40
Width [mm]	26
Mid-thickness [mm]	2.275
Keystone angle [degrees]	0.44
Minimal $I_c$ @ 4.22 K and 12 T [kA]	58.9
Minimal $I_c$ @ 4.22 K and 15 T [kA]	29.4
$I_c$ for extracted strands, 12 T and 15 T	> 90 % of virgin strand
RRR after full reaction	120

As shown by Table 1, the critical current target at 4.2 K was taken to be quite ambitious: 1636 A (12 T) and 818 A (15 T). These currents respectively correspond to critical current densities in the non-copper part of 3000 and 1500 A/mm<sup>2</sup>. These very high critical current values are needed to achieve a 14-15 T magnetic peak field in large aperture Nb<sub>3</sub>Sn superconducting magnet. According to the adiabatic stability criterion [4],  $J_c \cdot d_{eff}$  is a figure of merit for stability and should be small to avoid magnetic instabilities (flux jumps).  $d_{eff}$  is the effective filament diameter and can generally be approximated by the diameter of the reacted Nb<sub>3</sub>Sn area, i.e. the size of the sub-element (SE) as limited by the diffusion barrier. The effective diameter, which should be kept as low as possible, was chosen to be less than 50 μm for NED strand. One should mention that the specified 50 μm value relates to the effective filament diameter as determined from the strand design, i.e. the SE diameter before heat treatment. To reach such a low effective filament diameter, the number of SEs has to be quite high for a 1.25 mm strand diameter (in the range of 250-300), which could induce fabrication problems. As an example, a wire with 54 SEs (~ 110 μm equivalent diameter for 1.25 mm strand) and with a critical current density exceeding 3000 A/mm<sup>2</sup> at 4.2 K and 12 T is commercially available in the US [5]. However, when assembling 198 SEs, workability problems were encountered and the critical current density was reduced by ~ 10 % as compared to the 54 SE strand mentioned above [5]. Thus, the target of NED, i.e. a 1.25 mm strand, 3000 A/mm<sup>2</sup> in the SE at 4.2 K and 12 T and 50 μm SE diameter, is a very challenging one. In addition to workability issues, when augmenting the number of SEs, the SE barriers are getting thinner and generally more distorted; their integrity then becomes more doubtful. This is especially true for wires deformed during the cabling. During the long reaction heat treatment necessary to achieve high critical current density Nb<sub>3</sub>Sn conductors, the Residual Resistance Ratio (RRR) of the copper stabilizer can sharply decrease due to likely tin contamination of the copper through the damaged barrier, thus dampening the dynamic stability of the conductor [6] and the magnet protection. Therefore the problem of sub-element barrier integrity appears to be a crucial issue for NED project. This is the reason why deformation studies are systematically performed on NED strands. Although such flat deformation done by means of rollers is not equivalent to the more complex real cabling deformation which incorporates twisting of the wires on the cable edges, these studies, including micrograph examinations and RRR measurements, can be at least semi-quantitatively used for probing the relative mechanical behavior of the strand to cabling deformation without waiting for cabling trials which remain the ultimate test. In parallel, a finite element mechanical model [7]-[8] was launched as well to assess the SE and barrier integrity and to allow for selecting the best strand design parameters.

At last, one should mention that, among the various processes to produce Nb<sub>3</sub>Sn conductors, the bronze route, allowing for fine filament achievement, was ruled out due to critical current densities not exceeding ~ 1000 A/mm<sup>2</sup>. NED efforts were then focused on two fabrication methods: ITD and PIT.

### 3. Development current status for Alstom/MSA ITD wires

#### 3.1. Strand development

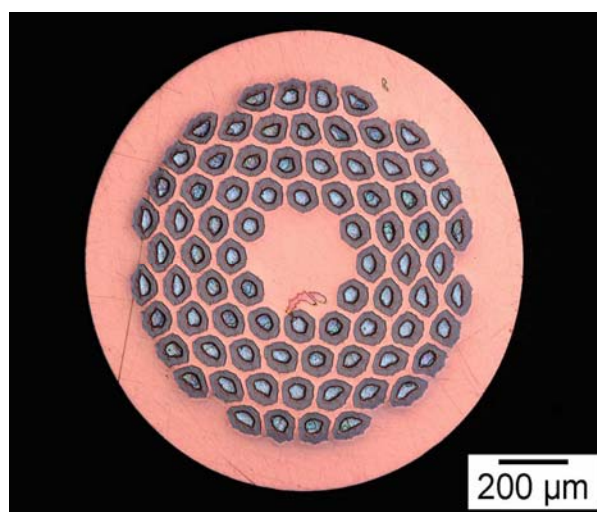
Before NED project, Alstom/MSA produced Nb<sub>3</sub>Sn wires with a relatively low critical current density in the non-copper part of ~ 800 A/mm<sup>2</sup> (4.2 K and 12 T) for ITER or CEA/Saclay quadrupole [9]. The CEA strand contains, for example, 19 SEs, ~ 100 μm in diameter and surrounded by a collective diffusion barrier. For NED project, more SEs with individual barriers are needed to achieve smaller diameter SEs. Fabricating such a wire represented undoubtedly a challenge as compared to previous Nb<sub>3</sub>Sn strands manufactured by Alstom/MSA. Therefore, during the first step of NED, MSA focused its efforts mainly on SE designs to ensure a successful cold drawing process. For this purpose, several SE configurations were launched, varying design parameters like the tin distribution for example. A strand, with 78 SEs containing more copper than necessary to achieve NED specifications, was successfully drawn down to the nominal diameter. This wire, called B1 and having a SE diameter of ~ 88 μm (instead of 50 μm), presents a critical current density in the non-copper part of ~ 1500 A/mm<sup>2</sup> at 4.2 K and 12 T, as expected from the strand design and as measured consistently by Alstom/MSA,

CERN and INFN-Milan. It is worth mentioning that, with this strand, Alstom/MSA practically doubled the critical current density of its previous CEA strand.

For the second step of NED, Alstom/MSA launched a strand presenting a design similar to that of B1 strand. However, this time, the SE contained enough niobium and tin to achieve a non-copper critical current density of 2300-2500 A/mm<sup>2</sup> at 4.2 K and 12 T. The characterization results of this strand will be detailed in the next subsection.

### 3.2. Strand characterization results

The strand, B2, contains 78 SEs composed of Nb filaments arranged around a tin core. In consequence, the SE diameter before heat treatment (HT) is 88 μm and is not fulfilling yet the specifications (< 50 μm), as already mentioned. A general cross-section view of the un-reacted strand at 1.25 mm diameter is shown in Fig. 1.



**Figure 1.** Cross section view of B2 strand (un-reacted) at nominal diameter (~ 1.25 mm).

Alstom/MSA has also drawn a B2 wire length down to 0.83 mm diameter in order to check its workability. Samples at both 1.25 mm and 0.83 mm diameters were delivered and characterized at CERN. The characterization results are summarized in Table 3.

**Table 3.** B2 strand characterization results (see text for more details)

Wire dia. [mm]	SE dia. [μm]	Cu/non-Cu	Radial expansion [%]	RRR	$I_c$ , 4.33 K, 12 T [A]	$J_c^{non-Cu}$ , 4.33K, 12 T [A/mm <sup>2</sup> ]	$J_c^{non-Cu}$ , 4.22K, 12T [A/mm <sup>2</sup> ]
1.254	88	1.59	~ 2	41	903	1896	1933
0.826	58	1.59	~ 2	6	432	2089	2129

The copper-to-non-copper ratio is 1.59 for both diameters and thus higher than the maximal specified value (1.35). Samples were prepared for critical current ( $I_c$ ) measurements at both diameters. The procedures for mounting such samples on VAMAS-type sample holders, heat treating and measuring them were already presented in [10]. The critical current criterion used for NED is 0.1 μV/cm. For both wires,  $I_c$  samples and RRR straight samples were heat treated under vacuum

conditions in Geneva University oven with the same following HT schedule: 48 h at 220 °C, 48 h at 410 °C and a final plateau of 50 h at 660 °C. The radial expansion following HT was observed to be ~ 2 % for both wires considered. The RRR values measured for 1.25 mm and 0.83 mm diameter samples are respectively 41 and 6, apparently indicating a tin contamination of the stabilizer. The  $I_c$ , as measured at CERN critical current facilities [11] and for 4.33 K and 12 T, is respectively 903 A and 432 A, for 1.25 mm and 0.83 mm strands, corresponding to a non-copper part critical current density ( $J_c^{non-Cu}$ ) of ~ 1900 and ~ 2090 A/mm<sup>2</sup> respectively. After temperature correction on the basis of Summers' fit formula [12], assuming a typical strain of 0.3 %, the  $J_c^{non-Cu}$  values, at 4.22 K and 12 T, are respectively ~ 1930 and ~ 2130 A/mm<sup>2</sup>. It is worth mentioning that the 0.83 mm sample presents thus a critical current density larger by ~ 10 % than that of the 1.25 mm strand. In order to explain this discrepancy, cross-sections of both samples were analyzed at CERN by means of SEM/EDS. It appears that, in average, the Nb<sub>3</sub>Sn phase is richer in tin for the 0.83 mm sample (24.3 ± 0.2 % at. Sn) than for the 1.25 mm wire (23.6 ± 0.2 % at. Sn). Moreover, more tin remains in the SE core of a 1.25 mm sample after reaction (6-7 % at. Sn) as compared to 0.83 mm sample (3-4 % at. Sn). Last but not least, the filaments of the 1.25 mm wire appeared to be not fully reacted, contrary to 0.83 mm sample. Therefore, it seems that the HT schedule is not optimized, at least in the case of the 1.25 mm diameter wire. The HT optimization is currently under investigation at Alstom/MSA. For the meantime, various HT schedules (with longer final plateaus) provided a  $J_c^{non-Cu}$  at 4.2 K of 1800-1900 A/mm<sup>2</sup> [13], i.e. fairly comparable to that measured at CERN for 50 h at 660 °C.

#### 4. Development current status for SMI PIT conductors

##### 4.1. Strand development

In the past, SMI produced PIT wires by stacking up to 504 tubes made of either Nb or Nb-Ta and comprising a core filled with a rich-tin powder [14]. The 504 filaments strand presented a filament diameter of ~ 20 µm for a 0.9 mm diameter wire. However, the  $J_c^{non-Cu}$  was relatively low (~ 1700 A/mm<sup>2</sup> interpolated value at 4.2 K and 12 T), in comparison to wires with less and bigger filaments. The best  $J_c^{non-Cu}$  value ever achieved by SMI before NED is ~ 2400 A/mm<sup>2</sup> at 4.2 K and 12 T, for the wire B179, 1 mm in diameter and 192 filaments of ~ 55 µm, corresponding to ~ 70 µm at 1.25 mm strand diameter.

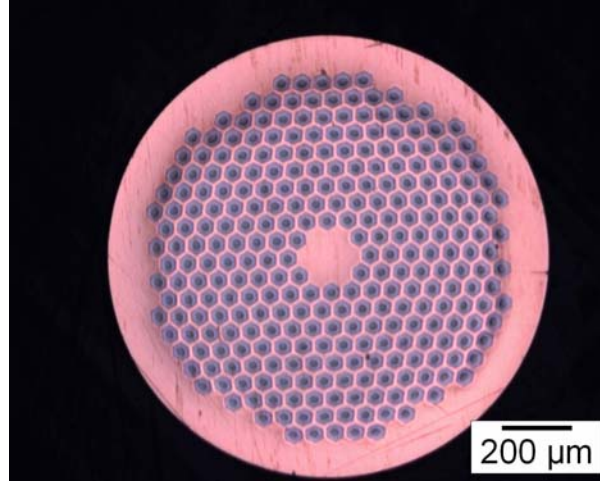
For the first step of NED, SMI focused on powder qualification, the target being to reach a  $J_c^{non-Cu}$  larger than 2500 A/mm<sup>2</sup> (4.2 K, 12 T) in a 1 mm diameter wire composed of 50 µm filaments. For this purpose, two wires, B201 and B205, were manufactured by SMI with a powder particularly enriched in tin. These wires include respectively 192 and 156 filaments and they are ~ 300 m long. However, during HT, both wires presented tin leaks probably due to high pressure following tin melting at ~ 230 °C and their  $J_c^{non-Cu}$  values, ~ 2100 A/mm<sup>2</sup> at 4.2 K and 12 T, were then lower than that measured for B179. Nevertheless, the B201 wire, including more interfilamentary copper than previous SMI strands, exhibited a much better mechanical behavior to flat deformation.

For the second step of NED, SMI fabricated two wires at nominal diameter (1.25 mm) and containing 288 ~ 50 µm diameter filaments. The SE spacing was kept similar to that of B201 wire. The characterization of these wires, B207 and B215, will be presented in the next subsection.

##### 4.2. Strand characterization results

The strand B207 was produced by SMI by incorporating 288 Nb-Ta tubes in the design. This strand has a copper-to-non-copper ratio of ~ 0.96, i.e. lower than specification. In the final strand, the filaments are 53 µm in diameter, as derived from micrographs. Following a reaction HT schedule of 84 hours at 675 °C, INFN-Milan measured the critical current at 4.24 K and 12 T for two samples. The  $I_c$  values were found to be consistent within 0.3 % and the average value is 1315 A, corresponding to a non-copper critical current density of ~ 2080 A/mm<sup>2</sup>. This relatively disappointing  $J_c^{non-Cu}$  can be explained by an inadequate heat treatment undergone by the powder before wire manufacturing [15]. For the same HT schedule mentioned, a high RRR value of 250 was measured for the B207 strand.

Afterwards, SMI fabricated a wire (B215) with a design similar to that of B207 (288 filaments) but with the appropriate Cu/non-Cu ratio and powder preparation. A single piece of  $\sim 1$  km long strand was produced. A general cross-section of the B215 strand at final diameter is presented in Fig. 2.



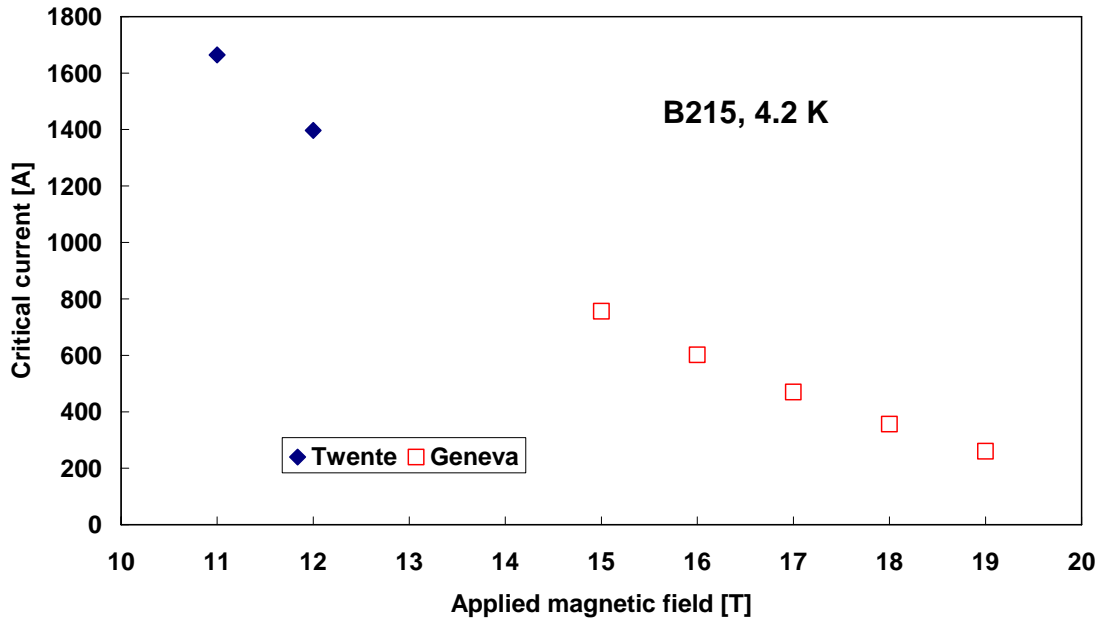
**Figure 2.** A cross-section view of the B215 wire at nominal diameter ( $\sim 1.25$  mm).

The filaments are indeed  $50 \mu\text{m}$  in diameter and the Cu/non-Cu ratio is within specifications, 1.22, as consistently measured by both CERN and Twente University. The characterization results of B207 and B215 are summarized in Table 4.

**Table 4.** Strand characterization results for SMI B207 and B215 strands (see text for more details)

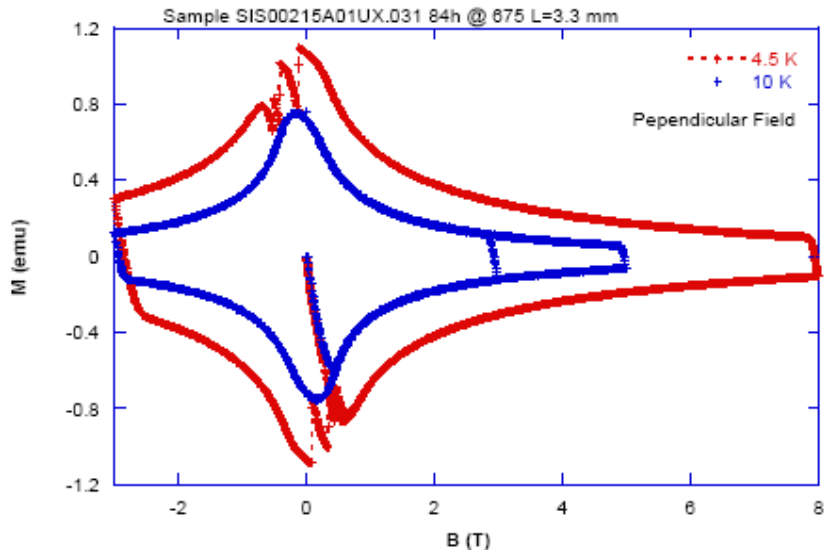
Wire	Wire dia. [mm]	SE dia. [ $\mu\text{m}$ ]	Cu/non-Cu	RRR	$I_c$ , 4.2 K, 12 T[A]	$I_c$ , 4.2 K, 15 T[A]	$J_c^{non-Cu}$ , 4.2 K, 12 T [ $\text{A}/\text{mm}^2$ ]
B207	1.255	53	0.96	250	1315	708	2084
B215	1.257	50	1.22	84	1397	756	2499

Following a HT schedule similar to that of B207 (84 hours at  $675^\circ\text{C}$ ), critical current measurements were performed on B215 samples at both INFN-Milan and Twente University. The  $I_c$  data were found to be consistent within  $\sim 3\%$ . The best  $I_c$  value measured is 1397 A (Twente), at 4.23 K and 12 T, and is  $\sim 15\%$  below NED target. This current corresponds to a  $J_c^{non-Cu}$  value of  $\sim 2500 \text{ A}/\text{mm}^2$  at the mentioned temperature and field conditions. Critical current measurements were also performed at Geneva University at higher applied magnetic fields and the  $I_c$  value measured at 15 T is 756 A, i.e. only 8% below NED target. This value corresponds to a non-copper critical current density of  $\sim 1350 \text{ A}/\text{mm}^2$  at 15 T. The complete  $I_c$  data, as measured for 4.2 K at Twente (11-12 T) and Geneva (15-19 T), are presented in Fig. 3. At last, some preliminary  $I_c$  measurements performed at CERN on a B215 sample at 1.9 K and 12 T showed an enhancement of the critical current of  $\sim 30\%$  as compared to 4.2 K and 12 T.



**Figure 3.** The critical current values ( $0.1 \mu\text{V}/\text{cm}$  criterion) as measured for B215 wire at 4.2 K by Twente and Geneva universities and as a function of applied magnetic field.

Magnetization measurements were performed as well on B215 reacted samples by INFN-Genoa (see more details on measurement setup and procedures in [16]). Such VSM measurements are presented in Fig. 4, in transverse magnetic field and for 4.5 K and 10 K. As clearly seen in Fig. 4, 4.5 K measurements showed only a few flux jumps between  $-0.8 \text{ T}$  and  $+0.8 \text{ T}$ .



**Figure 4.** The magnetic moment of a B215 reacted sample as measured by means of VSM and as a function of the magnetic field, at both 4.5 K and 10 K.



In addition, stability current measurements [17] were performed for 4.3 K at CERN. The stability current is the maximal current a strand can sustain without quenching, when sweeping the magnetic field at low values. For this purpose, the magnetic field was varied between 0 and 4 T at 0.3 T/minute ramp with a constant transport current of 2000 A (the power supply current limit at CERN); no quench was observed. Therefore, this high stability current, exceeding 2000 A, and the limited amount of flux jumps observed during magnetization measurements (Fig. 4) seem to clearly indicate a fair stability of the B215 strand against flux jumps.

#### 4.3. First cabling trials

The promising SMI wire, B215, was used to produce a Rutherford-type cable. Indeed, cabling trials were performed at Lawrence Berkeley National Laboratory (LBNL). ~ 900 m long wire was thus used to manufacture four short 40-strand cable lengths with various dimensions. The dimension ranges of all these cables, as measured at LBNL, are summarized in Table 5, together with the dimensions of a given cable (cable C) which was selected for assessing the critical current degradation due to cabling.

**Table 5.** Dimensional parameters for cable lengths produced by LBNL

Parameter	All cable lengths	Cable C
Finished length [m]	1.8-2.8	2.8
Average Mid-thickness [mm]	2.285-2.317	2.317
Average Width [mm]	26.922-26.988	26.978
Average Keystone angle [°]	0.392-0.415	0.394
Transposition length [mm]	150-191	150

Cable C has a filling factor of ~ 84 % before reaction and it is 2.8 m long. Strands were extracted from this cable and were distributed to CERN, INFN-Milan and Twente University for  $I_c$  sample preparation, varied HT schedules and measurements. These measurements are currently underway. However, preliminary critical current measurements performed at CERN, on three extracted strands and for a HT schedule of 120 hours at 650 °C, showed a reasonable  $I_c$  degradation of 4-8 %, as compared to virgin strand samples heat treated in the same batch, for 4.3 K and 12 T. This degradation is less than the maximal 10 % requested by NED specifications (see Table 2). In addition, RRR values for extracted strands, ~ 90, are similar to that of virgin samples. Therefore, even if these results have to be confirmed by other institutes, the cabling trials appear to be very successful for SMI/PIT B215 strand.

## 5. Conclusions and perspectives

In this work, the current status of NED conductor development has been briefly presented for both contractors. The two strand manufacturers have achieved substantial progress.

Alstom/MSA has developed a strand with a non-copper critical current density of ~ 2100 A/mm<sup>2</sup> at 4.2 K and 12 T. A new strand, currently in fabrication, is expected to achieve 2300-2500 A/mm<sup>2</sup> at the mentioned temperature and field conditions.

SMI has succeeded to produce a PIT strand with a critical current of ~ 1400 A at 4.2 K and 12 T, corresponding to a non-copper critical current density of ~ 2500 A/mm<sup>2</sup>. This wire contains 50 µm diameter filaments as requested. One should mention that the high critical current and the fine dimension filaments achieved for this wire constitute a world record for such a critical current density level. Moreover, magnetization and stability current measurements demonstrated the wire stability against flux jumps. Last but not least, cabling trials for this SMI/PIT strand, carried out at LBNL, were

successful. Preliminary critical current measurements on extracted strands indeed showed a moderate cabling degradation of 4-8 %. SMI has thus completed its R&D phase and it will shortly begin to produce the final strand for NED cable.

CERN is currently launching an ambitious high-field accelerator magnet program. The NED project and its results will constitute a base for this new R&D program.

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