LBNL Test Facility Dipole Project

Wire Specification

For the Production Cables

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**Revision History**

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# SCOPE

The objective of this specification is the acquisition of long lengths of superconducting wire with uniform properties for the Test Facility Dipole (TFD) production and spare coils. Property uniformity along the continuous strand length from any billet, as well as consistency of properties across different billets, thus directly relates to performance and uniformity of the cable. Margin for degradation during cable fabrication and other losses are accounted for by the specified parameters. The strand procurement seeks characteristics in manufactured Nb3Sn composite superconducting strands that exemplify the best present state of the art.

# APPLICABLE DOCUMENTS

The following documents apply to this specification. The document version in effect on the date of invitation to quote shall be the version in force for the purposes of defining and complying with this specification.

1. *TFD Material Naming Scheme*
2. International Standard IEC 61788-2: *DC critical current of Nb3Sn composite superconductors*
3. International Standard IEC 61788-11: *Residual resistance ratio of Nb3Sn composite superconductors*
4. International Standard IEC 61788-12: *Copper to non-copper volume ratio of Nb3Sn composite superconducting wires*
5. ASTM B170: *Standard Specification for Oxygen-Free Electrolytic Copper—Refinery Shapes*
6. ASTM B392: *Standard Specification for Niobium and Niobium Alloy Bar, Rod, and Wire*
7. ASTM B393: *Standard Specification for Niobium and Niobium Alloy Strip, Sheet, and Plate*
8. ASTM B884: *Standard Specification for Niobium-Titanium Alloy Billets, Bar, and Rod for Superconducting Applications*
9. ASTM F86: *Standard Specification for Oxygen-Free Copper in Wrought Forms for Electron Devices*

# NOMENCLATURE

This table defines acronyms listed in this document:

|  |  |
| --- | --- |
| Term | Definition |
| ASTM | American Society for Testing and Materials, a standards organization |
| *I*C | Critical current |
| IEC | International Electrotechnical Commission, a standards organization |
| LBNL | Lawrence Berkeley National Laboratory |
| Non-Cu | The portion of the conductor cross section contained within diffusion barriers, including the barriers themselves. This portion contains the superconducting Nb3Sn after reaction. |
| RRR | Residual resistance ratio, the ratio of resistance measured at room temperature to that measured just above the superconducting transition in zero magnetic field |

# TERMINOLOGY

Terminology from *TFD Material Naming Scheme* applies. *In addition*, the following terms apply to throughout this document:

## ***Sub-element****:* A self-contained unit that contains copper, tin, niobium, and possibly other elements that is intended to produce the Nb3Sn phase after a reaction.

## ***Lot:***All material having the same parent ingot or billet and having been annealed in the same furnace charge and processed on the same manufacturing line during the same production period.

## ***Non-copper***: The portion of the conductor cross-section that is composed of sub-elements not made from pure copper.

## ***Diameter*:** The diameter *d* is given by the arithmetic average of multiple simultaneous measurements of diameter for *n* axes perpendicular to the wire axis, . For a 2-axis measuring device reporting measurements *a* and *b* along orthogonal directions transverse to the wire axis, *d* = (*a* + *b*) / 2.

## ***Ovality****:* The ovality *o* is given by *o* = |*a – b*|, where *a* and *b* are the maximum and minimum dimensions measured along orthogonal directions transverse to the wire axis. A perfectly round wire thus has an ovality of 0.

## ***Unit Length*:** The length of each strand in a cable used to wind magnet coils.

## ***Yield***: The yield of a billet shall be defined as the ratio of *the total mass of pieces from the billet that meet all delivery requirements* to *the starting billet mass that can be processed in the “stable zone”*. The supplier shall state the 100% yield mass in a Disclosure Statement.

# PHYSICAL REQUIREMENTS AND PHYSICAL PROPERTY TESTING

## Strand Diameter:

### The strand diameter shall be determined by a calibrated multi-axis laser micrometer, with simultaneous measurement axes perpendicular to the strand axis. The requirement above is graphically presented in figure 1, for target diameter *d0* = 1.100 mm and tolerance  = 0.003 mm.

### The measurements shall be carried out at room temperature.



Figure 1. Graphical representation of multi-axis diameter measurements. The strand cross-section at a particular location is represented in blue.

### For each axis *i*,*,*measurements *di* shall be simultaneously recorded at least once per meter.

### The average and standard deviation *di* for all points acquired over the length of the piece shall be reported for each axis *i*.

### For each axis *i*, average values must satisfy 1.097 mm   1.103 mm.

### The strand diameter *d* (see 4.4) and the ovality *o* (see 4.5) shall be determined from the average values and the resulting determinations shall be reported.

### Supplier should note that, while it is possible to determine a strand diameter that meets the target diameter and tolerance by taking an arithmetic average that includes values which do not comply with 5.1.5, acceptance of the strand will be determined by both 5.1.5 and 5.1.6.

### Supplier shall also note that the maximum ovality value is 0.006 mm.

## Superconductor after reaction:

### The superconductor shall be formed by reaction at high temperature, after cabling and magnet winding have been completed.

### The superconductor shall be primarily Nb3Sn.

### The superconducting phase shall be alloyed with up to 2% Ti by atomic fraction in order to enhance the high field performance.

## Conductor materials and construction prior to reaction:

### The superconductor shall be formed within sub-elements that contain copper, niobium, tin, and other requirements to form the alloyed superconductor as in 5.2.3.

### The sub-elements (see 4.1) shall be independent and self-contained and surrounded by individual diffusion barriers. Strand designs that use a single diffusion barrier around a collection of sub-elements, or intend sharing of reactants between sub-elements, are not acceptable.

### The sub-element count shall be equal to 162.

### Supplier shall notice that this count defines an average sub-element diameter in micrometers, determined by the expression 1100 [*N* (1 + *R*)]–0.5, where *N* is the number of sub-elements and *R* is the measured copper to non-copper ratio per 5.4. The average defined by a count of 162, a diameter per 5.1.1, and *R* per 5.4.1 is thus 63 µm.

### Supplier shall have liberty to designate material grade and other conditions of sub-element raw materials, such as grain size, hardness, annealing temper, and so on to suit the manufacturing process.

### Use of Grade 1 niobium, per ASTM B392: *Standard Specification for Niobium and Niobium Alloy Bar, Rod, and Wire* or ASTM B393: *Standard Specification for Niobium and Niobium Alloy Strip, Sheet, and Plate*2.8 is strongly recommended.

### The sub-elements shall be surrounded by copper stabilizer. The stabilizer shall be oxygen free high conductivity grade (also known as electrolytic grade) copper per ASTM B170: *Standard Specification for Oxygen-Free Electrolytic Copper—Refinery Shapes*. Copper tubes, stacking elements, shims, and any other pieces intended for stability shall also be able to meet ASTM F86: *Standard Specification for Oxygen-Free Copper in Wrought Forms for Electron Devices*, class 2 or better, after component fabrication and prior to billet assembly.

### A central area of at least 2.5% of the strand cross section shall be copper stabilizer. Manufacturer shall note that 2.6% is equivalent to 7 of 169 elements as an example of a stacking pattern with hexagonal symmetry.

### Supplier shall provide photo-micrographs of the cross-section of final size wire sufficient to distinguish all assembly details.

## Copper Fraction and Copper to Non-Copper Volume Ratio:



Figure 2. Schematic describing the location of verification samples for each billet and additional verification samples associated with wire breaks.

### The copper volume fraction of the conductor shall not be less than 44.4 % over any portion of wire.

### The copper to non-copper ratio *R* shall satisfy 0.8  *R*  1.0.

### International Standard IEC 61788-12: *Copper to non-copper volume ratio of Nb3Sn composite superconducting wires*, or Appendix B of that standard, or similar methods to be accepted by the procurer, shall be used to determine the copper to non-copper volume ratio.

## Stand Twist:

### All strands shall be twisted such that all sub-elements follow the required rotation.

### The strand shall be twisted before the final sizing die.

### The twist pitch *p* shall satisfy 16.0 mm  *p*  22.0 mm.

### The twist shall be a right-handed screw with respect to the wire axis.

### The presence, pitch, and direction of the strand twisting shall be verified by light acid etching and microscopy observation, or other established methods to be accepted by the procurer. Photographs of these observations shall be included in the supplier quality control (QC) data package submitted for shipment approval.

## Length of strand pieces:

### Wire shall be delivered in integer multiples of 410 m (50% of total required units) and 425 m (50% of total required units). In addition, to ensure quality and homogeneity, the maximum acceptable number of pieces (as a result of breakages plus intentional cuts) per billet is ten. Supplier may ask for acceptance of billets with higher number of breakages and cuts. The purchaser has the discretion to decide whether to accept or not.

### Supplier shall understand that very long strand piece lengths are highly desirable, not only to reduce waste and excess, but also because this is a sign of high quality and homogeneity, both of the strand and of its manufacturing process.

### Strand pieces for QC purposes shall be nominally 20 m length and equally divided for supplier QC and purchaser verification QC testing (i.e. 10 m + 10 m).

### Any QC sample piece must be adjacent to one end of a delivered piece of production conductor or another QC sample piece. The verification sample piece delivered to the purchaser shall, moreover, be adjacent to the QC sample piece selected by the supplier, i.e. they should be cut from the same end of a long conductor piece. This is described in figure 2.

### Supplier shall not make unnecessary cuts to continuous pieces.

### Supplier shall report the manufacturing yield for each billet per 4.7. When reporting yield, supplier shall furthermore report a compilation of all final-size pieces derived from the billet, listing length, diameter, and either the mass of the piece or a conversion of mass per unit length for each piece.

## Surface Condition:

### Each strand piece length shall be checked continuously on its overall length by an eddy-current method to detect inclusions, voids, cracks, and other surface defects.

### The strand surface at the final diameter shall be free of any surface defects, slivers, folds, gouges, laminations, or inclusions, and shall not have any component other than the copper stabilizer material visible.

# ELECTRICAL AND MAGNETIC REQUIREMENTS AND ELECTROMAGNETIC TESTING

## Critical Current:

### The strand critical current *Ic* shall exceed 600 amperes at 16 T field and 4.2 K temperature.

### The strand critical current *Ic* shall exceed 760 amperes at 15 T field and 4.2 K temperature.

### The strand critical current *Ic* shall exceed 570 A at 16 T, 4.2 K, and also exceed 720 A at 15 T, 4.2 K, after rolling to 0.935 mm thickness (i.e. 15% reduction).

### The *Ic* shall be measured according to International Standard IEC 61788-2: *DC critical current of Nb3Sn composite superconductors* for fully reacted strands at the final diameter.

### The *Ic* shall be defined for an electric field criterion of 10 μV/m.

### The *Ic* shall be reported without any self-field correction.

### The voltage tap separation for determining *Ic* shall be at least 40 mm.

### The *Ic* test at 16 T and 15 T, 6.1.2, shall include the measurement of *n* as defined in International Standard IEC 61788-2: *DC critical current of Nb3Sn composite superconductors*International Standard IEC 61788-2: *DC critical current of Nb3Sn composite superconductors*. The value of *n* shall equal or exceed 30. The electric field range used to determine *n* shall be at least one decade in magnitude and shall include the *Ic* criterion.

### Supplier shall note that the international standard requires reporting of uncertainty, which includes variations of liquid helium bath temperature from nominal 4.22 K, magnetic field uncertainty, and calibrations.

## Residual Resistance Ratio, RRR:

### The value of *RRR* shall exceed 150 for final size strand after full reaction.

### Manufacturer shall note the reaction conditions for all samples must comply with 8.1.7.

### In addition, the value of *RRR* shall exceed 100 for a final size strand rolled to a thickness of 0.935 mm, i.e. 15% reduction, also after full reaction.

### International Standard IEC 61788-11: *Residual resistance ratio of Nb3Sn composite superconductors* shall be used to determine the RRR value.

### Supplier shall note that RRR is defined as the ratio of resistance at 293 K (room temperature) to that defined by the intersection of the superconducting transition region and the onset of the normal conducting region upon warming the sample from the superconducting state. Measurements at 20-22 K *may* be accepted for the low-temperature resistance only after validation proof in the form of benchmarking exercises involving LBNL or FNAL or their subcontractors.

### Manufacturer shall note that the international standard accommodates convenient ambient measurements at 20 ± 10°C via a correction formula. Manufacturer also shall note that thermometry need not be used for the cryogenic measurement if the international standard is followed properly.

## Magnetization

### Magnetization data are welcomed but not required in the QC data package submitted for shipment approval.

### A strand design that exhibits flux jumps in perpendicular magnetic fields above 2.7 T at 4.2 K is not acceptable.

### A vibrating sample magnetometer, extraction magnetometer, or SQUID magnetometer shall be used for the magnetization measurement. Supplier shall notice that many instruments report magnetic moment in emu (electromagnetic units) instead of A m2. Supplier shall take careful note of any conversions used; the conversion from emu to A m2 is to divide emu by 1,000.

# MECHANICAL REQUIREMENTS AND MECHANICAL TESTING

## Sharp bend test

A strand at final diameter in the un-reacted state shall withstand a bend that folds the wire back upon itself lengthwise without cracking or rupture. Upon etching the copper from the bent region, no broken sub-elements shall be visible.

## Strand Spring-Back:

### Final diameter strand shall not spring back more than 720 arc degrees.

### A 10-turn, 10 mm diameter helix shall be wound with 20 N tension and the ends shall be marked. Upon removal of tension, the helix will unwind. The angular rotation of the marked ends during unwinding of this spring is defined as the spring-back.

# HEAT TREATMENT REQUIREMENTS

## Schedule

### The manufacturer shall recommend a heat treatment (HT) schedule based on internal verification tests. The schedule shall be chosen to ensure performance of delivered pieces meets or exceeds the required specifications after reaction, as well as meet the other constraints below. Once approved by purchaser, the HT schedule shall not change for all materials delivered except by written notification from the purchaser.

### The total duration of the heat treatment, from the time when temperature starts its first increase to the time at which the furnace is turned off and allowed to cool from high temperature, shall not exceed 300 hours.

### The heat treatment time during the high temperature reaction segment, during which time the Nb3Sn is formed, shall not be less than 40 hours.

### The heat treatment shall not incorporate temperature increase at a rate faster than 50°C per hour. Supplier shall take note that finished magnets have large mass, which prohibits rapid thermal cycles. Temperature ramp rates shall not affect the final performance.

### The maximum temperature shall be as low as possible, yet sufficient to guarantee performance and to ensure the Cu-Sn alloy left in the subelement at the end of the reaction heat treatment will comprise α-phase solid solution only.

### The recommended heat treatment environment shall be flowing Ar at atmospheric pressure or vacuum.

### Any HT performed by the manufacturer for purposes of verification testing must be identical to the HT recommended for delivered product. This includes exposure to the heat treatment environment for all samples; special shielding or other provisions will invalidate the supplier QC tests unless explicit written agreement has been given by the purchaser.

## Uncertainty

Manufacturer’s HT equipment shall comply within the following uncertainty:

### All thermocouples used for recording the sample temperature must be calibrated to within ±1°C at the maximum HT temperature

### Actual thermocouple readings at sample positions shall be within of 2 °C of each other.

### The average of all thermocouples must be within ±5°C of the target temperatures for any dwell stage.

### The dwell time, from the point in time when the average temperature reaches within ±5°C of the temperature dwell target, until the point in time that the temperature falls outside ±5°C of the dwell target, must be within ±2 h of the target dwell time.

# other TESTING AND REPORTING REQUIREMENTS

Supplier shall submit a quality plan that includes testing to verify compliance with this specification. The following items are required elements of the plan.

## Raw materials

Supplier shall verify the following properties for each lot received: copper RRR, niobium RRR, niobium mechanical properties, niobium microstructure, and chemical assay for all components.

## Minimum sampling rate for conductor physical and electromagnetic property tests

A schematic of the *minimum* sampling rate for each wire billet is shown in the Tables in 9.3. In general, strand pieces for verification testing shall be taken from production pieces at the extremities of the billet (the “P” and “T” locations, sometimes called “front end” and “back end”).

## Minimum testing rate for properties

The required testing rate at the start of production is specified in the following sections. Purchaser may designate a different rate of testing at later times, depending on the progress of production. A production process with good statistical controls is highly desirable, because this should facilitate a reduction of testing frequency at later stages.

### Conductor physical property testing

|  |  |
| --- | --- |
| Parameter or characteristic | Testing rate |
| Strand diameter | Each piece, see 5.1 |
| Ovality | Each piece, see 5.1 |
| Twist Direction and Pitch | Each piece |
| Length | Each piece |
| Strand Spring Back | 6 per billet (3 point, 3 tail) |
| Sharp bend test | 6 per billet (3 point, 3 tail) |
| Cu : Non-Cu volume Ratio (*R*) | 2 per billet |
| Photomicrographs | 1 per billet |

### Electromagnetic property testing of round wires

|  |  |
| --- | --- |
| Parameter or characteristic | Testing rate |
| Critical current | 2 per billet (see also 9.4 below)  15 and 16 T, 4.2 K (required) |
| *n*-value | (with tests above; 15 T and 16 T required) |
| Residual Resistance Ratio RRR | 2 per billet (see also 9.4 below) |
| ~~Magnetization\* at 3 T, 4.2 K~~ | (Not required) |

### Electromagnetic property testing of rolled wires

Supplier shall conduct tests for verification pieces rolled to 0.935 mm thickness (i.e. 15% reduction). Supplier shall place the flat surface of the rolled sample against the test mandrel when conducting electromagnetic property testing.

|  |  |
| --- | --- |
| Parameter or characteristic | Testing rate |
| Critical current | 1 per billet  15 and 16 T, 4.2 K (required) |
| *n*-value | (with tests above; 15 T and 16 T required) |
| Residual Resistance Ratio RRR | 1 per billet |

## Additional testing for billets with more than 2 pieces

Supplier shall provide the additional verification tests below for internal pieces of billets that have 3 or more pieces for delivery.

|  |  |
| --- | --- |
| Parameter or characteristic | Testing rate |
| Critical current | If *I*C (16 T, 4.2 K) ≥ 625 A and RRR ≥ 200 for both tests under 9.3.2, then one additional test should be performed on the piece immediately after the first break or cut location. Otherwise, all pieces for delivery should receive tests. |
| *n*-value | (with tests above) |
| Residual Resistance Ratio RRR | If *Ic* (16 T, 4.2 K) ≥ 625 A and RRR ≥ 200 for both tests under 9.3.2, then one additional test should be performed on the piece immediately after the first break or cut location. Otherwise, all pieces for delivery should receive tests. |

# IDENTIFICATION REQUIREMENTS

All delivered conductor pieces shall comply with *TFD Material Naming Scheme*.. In addition, all pieces kept at the supplier for validation shall be labeled according to the same scheme to facilitate cross-referencing.

Supplier shall notice that the scheme for a piece contains the billet number and an identifier of the piece(s) that result from wire drawing. If the billet number does not conform to the naming scheme, supplier and purchaser shall determine a number to comply with the 14-character scheme.

Supplier shall notice that samples used for verification of properties receive additional identification under the naming scheme.

# DOCUMENTATION AND DELIVERY REQUIREMENTS

Supplier shall note that this specification is part of a larger acquisition plan, which compels all suppliers to provide certain information as part of their offer. The information includes a management plan, a quality assurance plan, a manufacturing plan, and evidence of qualification. The details of the acquisition plan are not discussed here.

Supplier shall disclose the design of the conductor (including a list of critical raw material properties) and a list of the significant subcontractors in a Disclosure Statement.

All test results shall be supplied in a format mutually agreed by the supplier and LBNL. Raw data, certificates of materials, processing records, and similar information related to the production of strand shall be made available after a reasonable period upon request by LBNL.

## Documents required for Request for Delivery Approval

Supplier shall *not* deliver any product without authorization or approval from the purchaser. Supplier shall schedule deliveries based on billet number or production lot number.

Prior to the shipment of a product for delivery, the supplier shall notify the purchaser in writing of intent to ship. The notification shall include the following information:

* The billet pedigree and piece report;
* Certificate of conformity to specification for each billet;
* Raw materials certifications;
* Test results and test reports, using the standards or test methods specified herein.

In addition, the supplier shall be prepared to transmit the raw data from tests, micrographs or microscopy analyses, or other raw information related to the purchaser’s database. Purchaser may request this data in conjunction with the strand delivery, or as a follow-up, as deemed appropriate.

## Authorization to Deliver

The purchaser will compare the supplier’s information with information obtained from benchmarking validation exercises. Any discrepancies will be resolved to the satisfaction of the purchaser on a case-by-case basis. Purchaser may choose to waive some or all product validation tests associated with a particular billet.

Purchaser shall issue a written authorization to deliver as an indication of product acceptance.

## Shipping Instructions for Long Pieces

### Long strand pieces shall be shipped on spools. Each spool shall contain only one piece of strand. No preference is given to the spool axis.

### The spools shall be labeled with:

* Date of shipment,
* The Purchase Order Number (with Shipment identifier where appropriate),
* The Billet number and the Production Lot identifier (if any);
* The 14-character virgin strand identifier in characters and in code-39 barcode (see *TFD Material Naming Scheme*);
* The length of the piece;
* The spool net and tare weights;
* The direction of wire drawing relative to the end.

Supplier shall affix this information directly to the outside of the spool.

### Packaging

The outside end shall be secured to the spool, such as by wrapping with a plastic shrink wrap.

If the spool is packaged in a cardboard box, an additional copy of the spool label should be affixed to the outside of the box and the spool must be protected against water and handling. The strand shall be shipped in the un-reacted condition, preferably in sturdy wooden crates. Cardboard boxes can be shipped securely on a wooden pallet provided that the dimensions of the pallet prevent shipping damage.

## Shipping Instructions for Verification Pieces

Pieces supplied to the purchaser (or its subcontractor) for validation shall be packaged in sealed plastic bags. Only one piece per bag shall be permitted.

The bag shall be labeled with the virgin strand identifier in characters and in code-39 barcode, with the appropriate suffix that identifies a validation piece, as well as the purchase order numbers (with shipment identifier where appropriate). See *TFD Material Naming Scheme*.

Pieces shall be shipped in the un-reacted state.

Multiple bags can be shipped together.

All shipment shall use protective cartons or boxes.

# ANNEX – SUMMARY OF PERFORMANCE REQUIREMENTS

|  |  |  |
| --- | --- | --- |
| Parameter or characteristic | Value | Unit |
| Superconductor composition | Ti-alloyed Nb3Sn |  |
| Strand Diameter | 1.100 ± 0.003 | mm |
| Critical current at 4.2 K and 16 T | > 600 | A |
| Critical current at 4.2 K and 15 T | > 760 | A |
| *n*-value at 16 T | > 30 |  |
| Count of sub-elements  (Equivalent sub-element diameter) | 162  (≤ 63) | (µm) |
| Mean Cu:Non-Cu volume Ratio  Variation around mean  (Mean copper fraction) | 0.9  ± 0.1  (47.4%) |  |
| Residual Resistance Ratio *RRR*  for reacted final-size strand | ≥ 150 |  |
| Twist Pitch | 19.0 ± 3.0 | mm |
| Twist Direction | Right-hand screw |  |
| Strand Spring Back | < 720 | arc degrees |
| High temperature HT duration | ≥ 40 | Hours |
| Total heat treatment duration from start of ramp to power off and furnace cool | ≤ 300 | Hours |
| Heat treatment heating ramp rate | ≤ 50 | °C per hour |
| Rolled strand (0.935 mm thickness) critical current at 4.2 K and 16 T | > 570 | A |
| Rolled strand (0.935 mm thickness) critical current at 4.2 K and 15 T | > 720 | A |
| Rolled strand (0.935 mm thickness) *RRR* after reaction | > 100 |  |