

SUPERCONDUCTING  
SUPER  
COLLIDER

## MATERIAL SPECIFICATION

NO. SSC-MAG-M-401

TITLE: NbTi SUPERCONDUCTOR WIRE  
FOR SSC DIPOLE MAGNETS

ISSUE DATE 6-9-87REV. NO. 1REV. DATE 8-17-87

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## REVISION RECORD

REVISION NO.	DATE	PAGE	SUBJECT	APPROVAL
1	8/17/87	6 5  6 3 12 14 15	Delete "mid" in test #9 R <sub>295</sub> max I - 565 0 - 820 R <sub>10</sub> max I - 6.8 0 - 9.2 Twist pitch 2.0 ± 0.2 Delete "for 3 hrs. at 230°C" Use specific gravity of non-copper components Delete (duplicate of p.16) Delete (duplicate of p.19)	

1. Scope:

1.1 This specification establishes the requirements for the manufacture, inspection, test, identification and delivery of superconducting NbTi composite wire for use in SSC dipole magnets.

1.2 Wire Classification/Type - Wire controlled by this specification includes the following:

- SC 401 - Type A - Twisted - Annealed
- SC 401 - Type B - Twisted - Unannealed
- SC 401 - Type C - Not Twisted - Annealed
- SC 401 - Type D - Not Twisted - Unannealed

2. Applicable Documents:

The following documents of the issue in effect on the date of invitation to quote form a part of this specification to the extent specified herein:

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-109 - Quality Assurance Terms and Definitions
- CDA-101000 - Copper Development Association Standards
- SSC-MAG-M-400 - NbTi Alloy Bars and Rods
- MIL-C-12000 - Packaging
- ASTM E243-80 - Eddy Current Testing

3. Requirements:

3.1 Wire Material - Raw material used in the manufacture of SSC NbTi composite wire shall be procured to the applicable requirements of this specification and inspected/tested by the seller for conformance to those requirements before release for production use.

3.1.1 Nonconforming Raw Material - Material found to deviate from requirements shall not be dispositioned through any sell review process for production use without specific, prior buyer approval.

3.1.2 Raw Material Identification - Each lot of wire raw material shall be uniquely identified to allow all seller manufacturing, test, and inspection operation records for finished wire to be traceable to the affected material lot.

### 3.2 Technical Properties

- 3.2.1 Conductor Type: The conductor shall be a composite of NbTi filaments in an oxygen-free copper matrix. The superconductor composition shall be Nb 46.5 ± 1.5 wt.% Ti, and shall be high homogeneity grade or equivalent.
- 3.2.2 Critical Current: The conductors shall have a critical current greater than the values listed in Table I. These values refer to a test temperature of 4.222 K and a critical current criterion of  $\rho = 10^{-14}$  ohm · m, based on the total wire cross section area and with the applied magnetic field perpendicular to the wire axis. The currents given in Table I and the conditions defined above correspond to a current density in the superconductor of 2750 A/mm<sup>2</sup> at 5 T and 1100 A/mm<sup>2</sup> at 8 T.
- 3.2.3 Filament Size: The filament size shall be 6 microns. In order to insure that the filaments are electrically decoupled, the filament spacing shall be greater than 1.0µm in the billet design.
- 3.2.4 Copper-to-non-copper Ratio: The conductors shall have a copper-to-superconductor area ratio meeting the values specified in Table I.
- 3.2.5 Resistance at Room and Transition Temperatures: The resistance of the wire at room temperature (or normal state resistance is usually expressed as  $R_{295}$  or  $R_{295}$ ). It is an important parameter for magnet construction and depends on the content and purity of the copper. The resistance of the wire at transition temperature, usually expressed as  $R_{10}$  or  $R_{10}$ , can also provide a convenient independent check of the copper-to-superconductor ratio. The procedures for measuring  $R_{295}$  and  $R_{10}$  are described in this specification. The values for resistances and tolerances are given in Table I.
- 3.2.6 Copper Residual Resistivity Ratio: The RRR for wire at final size, equal to  $R_{295} / R_{10}$ , is defined by the values of  $R_{295}$  and  $R_{10}$  given in Table I. The target values for RRR as given there are greater than 83 for the inner layer wire and greater than 89 for the outer layer wire.
- 3.2.7 Twist Pitch: There are two options for twist pitch depending upon which cabling method will be followed.

Option A requires the wire to be twisted to produce a twist pitch of  $2.0 \pm 0.1$  twists/inch at the final wire size. All wire shall be twisted clockwise so that the filaments follow the same rotation as a right-hand screw thread. Requirements on twisting shall apply over the full length of delivered wire (no leaders with variable twist are allowed).

Option B requires that the wire be delivered in the untwisted condition.

- 3.2.8 Final Anneal: The wire may be ordered in the annealed or unannealed condition. If the wire is ordered in the unannealed condition, samples for testing purposes shall be annealed in order to verify that the  $R_{10}$  and RRR values are satisfied.
- 3.2.9 Surface Condition: The wire surface shall be free of all surface defects, slivers, folds, laminations, dirt, or inclusions. No NbTi filaments shall be visible.
- 3.2.10 Minimum Lengths: At least 90% of each order shall be delivered in lengths greater than 10,000 ft. The remaining 10% may be made up of lengths between 1000 ft. and 10,000 ft. If more than 10% of the ordered quantity falls below 10,000 ft. in length, the additional short lengths may be accepted, but at a reduced price which reflects the added costs associated with testing and cabling short lengths. Minimum length shall be determined after all lead and end defects have been removed by cropping. These defects include areas of distorted cross section due to wire point by swaging, and foreign material attached as a temporary leader, or areas of distorted filaments that occur at the start and end of an extrusion.
- 3.2.11 Mechanical Properties: The wire shall survive a sharp bend test without any visible sign of cracking at the outer diameter of the sharp bend.
- 3.3 Process Controls
- 3.3.1 Inspection/Test - The seller shall conduct those inspections and tests specified in Table I for each lot of finished wire at the cited frequencies, using the required material sample and test methods. In addition, the seller shall perform such other inspections and tests, and impose those process controls necessary to assure wire conformance with the technical requirements of this specification. In particular, detailed records shall be maintained for billet extrusion conditions (time and temperature of preheat, extrusion temperature and speed, post extrusion cooling, if any, etc.) and wire annealing conditions.
- 3.3.2 Nonconforming Material - In process or finished wire not meeting the requirements of Table I shall be identified and segregated from conforming items to preclude further processing or delivery to the buyer.
- 3.3.3 Product Identification - Wire lot identification shall conform to the requirements of specification SSC (SSC Wire and Cable Identification).

4. Quality Assurance Provisions

4.1 Quality Plan - The seller shall provide a documented description of the controls implemented to meet the requirements of this specification and any quality requirements specified in the ITQ or P.O. Any information of a proprietary nature must be identified in the seller's bid response. The seller will not be required to disclose this proprietary information, but will be required to show that adequate records and quality controls are maintained in these proprietary steps. The plan shall be submitted within 30 days of contract go-ahead and shall be considered acceptable unless disapproved in writing by the buyer.

4.1.1 Sampling - Where the seller proposes to use sampling in lieu of 100% inspection or tests, it shall be in accordance with the requirements of MIL-STD-105.

4.2 Certificate of Compliance - The seller shall provide a written statement certifying compliance with the requirements of the applicable P.O. and this specification with each product shipment.

5. Preparation for Delivery

5.1 Packaging - Spools shall be packed in accordance with the requirements of Level of MIL-C-2000 to assure that spools or wire are not damaged during shipment.

5.2 Reels/Spools

5.3 Winding Requirements - Wire shall be level wound.

5.4 Marking/Identification Requirements - Spools and exterior packaging shall be identified with the following information in the order shown:

"Superconducting Wire"  
Specification SSC-MAG-M-401 Type \_\_\_\_.  
Buyer P.O. NO. \_\_\_\_\_.  
Wire Identification No. \_\_\_\_\_.  
Length \_\_\_\_\_ feet.  
Weight \_\_\_\_\_ pounds.  
Source Billet Serial No. \_\_\_\_\_.  
Date of Manufacture \_\_\_\_\_.  
Name of Manufacturer \_\_\_\_\_.

TABLE I - PROPERTIES OF FINISHED WIRE  
TEST INSPECTION REQUIREMENTS

Examination or Test	Value I - Inner O - Outer	Test Specimen or Sample	Examination or Test Frequency	Test Method <sup>(1)</sup>
1. Critical Current				
Min at 5.6T	I-NA,0-285A	TM401-1	Each continuous	TM401-1
Min at 7T	I-328A @ 1.5/1		wire length	
2. Filament				
Size	6 microns		Both ends	
and Spacing	> 1.0 $\mu$ m	TM401-2	of each billet	TM401-2
3. Copper to Non-copper ratio	I 1.5 $\pm$ 0.1:1 O 1.8 $\pm$ 0.1:1	TM401-3	Both ends of each billet	TM401-3
4. Wire Diameter	I .0318 $\pm$ .0001 O .0255 $\pm$ .0001	Each 500 ft. mid and end	Each continuous wire gauging	Dimensional
5. Resistance	Values			
	micro $\Omega$ /cm	TM401-6	Each continuous	TM401-6
R <sub>295</sub> - Max	I - 565 O - 820		wire	
R <sub>10</sub> MAX	I 6.8 O 9.2			

(1) Test methods are included in Appendix A.

TABLE I - (continued)  
PROPERTIES OF FINISHED WIRE  
TEST INSPECTION REQUIREMENTS

Examination or Test	Value I - Inner O - Outer	Test Specimen or Sample	Examination or Test Frequency	Test Method <sup>(1)</sup>
6. Surface condition	No visible surface defects, slivers folds, laminations, dirt, inclusion or NbTi filaments.	100%	100%	Visual
7. Sharp bend Test	No visible cracking at sharp bend outer diameter.	TM 401-4	Each continuous wire	TM401-4
8. Springback Test	less than 980°-I 1090°-O	TM 401-5	Each continuous wire	TM-401-5
9. Twist Pitch (Type A & C wire only)	2.0 ± 0.2 clockwise twists per inch	Beginning and end of each continuous wire	Each continuous wire	Visual & dimensional gauging

(1) Test methods are included in Appendix A.

TABLE I - (continued)  
 PROPERTIES OF FINISHED WIRE  
 TEST INSPECTION REQUIREMENTS

Examination or Test	Value I - Inner O - Outer	Test Specimen or Sample	Examination or Test Frequency	Test Method <sup>(1)</sup>
10. Annealing (Type A & C) wire only)	R <sub>10</sub> Max I - 6.1 O - 9.0	Resistivity Ratio	Each continuous wire	TM401-6

(1) Test methods are included in Appendix A.

Appendix A - Superconductor Wire Test Methods

<u>Test Methods</u>		<u>Title</u>
No. 401-1	-	Critical current - short sample test method for critical current determination of twisted multifilament wire.
No. 401-2	-	Determination of filament size and billet spacing.
No. 401-3	-	Determination of copper to superconductor area ratio.
No. 401-4	-	Determination of sharp bend strength.
No. 401-5	-	Determination of springback properties.
NO. 401-6	-	Determination of normal state resistance of NbTi superconducting wire.

Test Method No. 401-1 - Critical Current

Short sample test method for critical current determination of twisted multifilamentary wire.

1. General Outline: Definition of Critical Current

The V-I curve is determined as a function of increasing current until an irreversible transition or quench occurs. This measurement is carried out in specified external fields, 5 T or 8 T typical, applied normal to the wire axis, and in a temperature bath of liquid helium at 4.222 K. For currents less than the quench current the V-I curve is reversible.

The critical current is defined as that at which the resistance per unit length, R, is:

$$R = 10^{-10} / (\pi D^2 / 4) \text{ ohms/m}$$

where D is the wire diameter in centimeters. The effective resistivity of the wire is  $10^{-14}$  ohm • m.

2. Sample Testing:

The seller shall measure the critical current for samples from each continuous length of wire at B = 5 T and 8 T, and T = 4.222 K. If a temperature of 4.222 K is not possible, measurements may be made at another

temperature and a conversion constant must be supplied. The conversion constant must be approved by Buyer. A 5-foot sample of wire adjacent to each length used by the Seller for critical current measurements shall be sent to the Buyer. These samples shall be identified by billet number, spool number, original continuous wire length, and purchase order number. Samples will be checked by the Buyer to insure that they conform to all aspects of the specification, both mechanical and electrical.

### 3. Sample Mounting

The sample wire is most conveniently mounted on a cylindrical former so that it fits in a solenoid magnet (see Section 4 below). Either bifilar or monofilar mounting arrangement may be used, if the procedures outlined below are followed. A non-inductive (bifilar) form will provide adequate length, reduce inductive voltage signals, and provide for ease of connection; see Fig. 1. Shorter, monofilar mounts may be used if adequately sensitive signal detectors are available; voltage taps are arranged as in Fig. 2 in this case. Means must be provided for constraint of mechanical motion without interfering with coolant contact: use of a G-10 former with grooved location of wire and careful tensioning during mounting. Care must be taken to ensure that a temperature gradient is not introduced into the region of measurement (gauge length). Care must also be taken in bending the samples, especially at the end of a bifilar sample.

### 4. Procedure

The sample length (between voltage taps) should be  $\geq 25$  cm. This corresponds, typically, to a voltage drop of several microvolts. This is readily measured with the aid of a suitable preamplifier or digital voltmeter. Samples of shorter length may be used if a well functioning nanovolt detection system is available. Equipment must be capable of determining the effective resistivity to a precision of 10%.

The amplifier signal should be recorded on an X-Y recorder (or if desired in a digital memory device). The V-I curve may be taken either point-by-point (current constant for each measurement) or continuously if induced signals due to ramping are not too large or noisy. Typically, current is supplied by a stable, well-filtered power supply. The current should be measured to a precision of  $\pm 5\%$ . Use of a low resistance normal metal shunt connected across the sample is permitted provided the resulting correction for shunt current is accurately known and is  $< 1\%$ . Electronic circuitry for quench protection is preferable. Frequently, a quality index,  $n$ , is estimated using the equation  $V = \text{constant} \times I^n$ .

### 5. Magnetic Field

The external field is most conveniently applied by means of a superconducting solenoid. The field must be uniform over the sample reference length to  $\pm 0.5\%$ . The direction between field and wire axis must be  $90^\circ \pm 6^\circ$  everywhere. This range of angles corresponds to a variation in  $I_c$  of 0.5%.

## 6. Temperature Bath Correction

The specification temperature is 4.222 K, that of boiling helium at standard atmospheric pressure. The bath temperature must be recorded with the aid of appropriate thermometry (cryogenic thermometer or vapor pressure of bath) with a precision of  $\pm 0.010$  K (10mK). Deviations of 25 mK or less from 4.222 K correspond to an error in  $I_c$  of 1% or less and may be ignored. For larger temperature excursions the "linear T" type of correction should be applied:

$$\frac{I_c}{I_m} = \frac{T_c - T}{T_c - T_m}$$

where  $T_c$  is the transition temperature at the specified magnetic field. ( $T_c = 7.2$  K at 5T and 5.7 at 8T.)  $I_m$  is the current measured at temperature  $T_m$ , and  $I_c$  is the critical current at the specification temperature,  $T (= 4.222$  K).

### Test Method No. 401-2 - Determination of Filament Size and Billet Spacing

Purpose: To determine the acceptability of filament size and billet spacing.

Method: Mount a suitably sized strand sample in an epoxy base and polish an appropriate cross section. Photograph at 1000x or higher and measure with metal scale calibrated in millimeters. Take ten measurements of filament size and spacing and average the results.

Test Method No. 401-3 - Determination of Copper to Superconductor Area Ratio.

COPPER TO SUPERCONDUCTOR RATIO TEST PROCEDURE

1. Materials List:      Deep Petri Dishes                      50% Nitric Acid Solution  
                                 Scale    Oven  
                                 Tweezers    Alcohol  
                                 Wire sample - 12" Length                      Sodium Bicarb
2. Label a clean petri dish the LBL number for each wire to be tested.
3. Wipe each 12" long wire sample with alcohol, coil and place in its assigned petri dish.  
Use tweezers to handle wire for the rest of testing procedure. Weigh the wire sample to the nearest 1000th of a gram and record its weight on the incoming strand inspection form.
4. Place 1 tabsp. of sodium bicarb in a container and dissolve in-water. Use this to neutralize any acid spills.  
DANGER - Nitric acid can cause severe burns and may be fatal if swallowed. Read all labels and use all safety precautions in handling acids.
5. Under fume hood, pour 50% nitric acid and 50% water into each petri dish to cover the wire samples. Place signs (WARNING NITRIC ACID).
6. Allow 30 min. then check each wire sample to see if all of the copper is etched away. This is done visually and by using tweezers to check for stiffness. Be careful not to cause damage to the sample. Stiffness indicates presence of copper. If copper is present, but acid appears to have stopped etching, pour the acid into a used acid container and add fresh solution.
7. After the copper is completely etched away, pour acid into a well labeled used acid container. The remains left in the petri dish are the superconductor filaments.
8. Gently rinse the filaments in the petri dish. Be careful not to rinse away loose filaments. Repeat 4-5 times or until thoroughly rinsed.
9. Put the petri dish containing the sample in the oven at 60°C - 70°C for approximately 15 minutes. Gently move the filaments to prevent sticking to the petri dish.
10. Dry the filaments for approximately 45 min.. Weigh the sample to the nearest 1000th of a gram and record weight on the incoming inspection form. With these weights, you can compute the copper-to-superconductor ratio.

11. Repeat steps 10 and 11. These two measurements should agree to within 5%.

12. Use the filament weight and copper weight in the following formula:

$$\text{Cu/SC, Ratio} = \frac{\left( \frac{\text{Weight of copper}}{\text{Sp. gr. of Cu}} \right)}{\left( \frac{\text{Weight of non-copper components}}{\text{Sp. gr. of non-copper components}} \right)}$$

Test Method No. 401-4 - Sharp Bend Test Procedure

The purpose of this test is to approximately simulate the deformation to the superconductor wire that may occur during cabling. The sharp bend fixtures are made to produce 20% deformation for each of the two wire diameters used. The fixtures are labeled accordingly

.0255 Dia.

Part No. 21M925 A-C

.0318 Dia.

Part No. 21M925 1-3

- 1) Cut a 3 inch sample of wire to be tested. Bend the sample in half and place the bend in the slot of the appropriate fixture.
- 2) Slide the mating top of the fixture in the slot and squeeze them together until closed using a bench vise.
- 3) Remove the top of the fixture and loosen the side screw.
- 4) The sample now resembles a hairpin. Examine the bend under a microscope to determine if the wire cracked or deformed in a way to inhibit cabling. This requires some knowledge of cabling and the limitation of sample deformation.
- 5) Etch the sharp bend sample in nitric acid. Use all precautions in handling acids. Examine the sample again to determine possible filament damage. This also requires some knowledge of filament characteristics.
- 6) Repeat this test a minimum of three times and fill in the inspection report as to whether the sample passed or not.

Test Method No. 401-5 - Determination of Springback Properties

1. Purpose

This test establishes a standardized method for testing superconducting (S.C.) wire to determine its springback acceptability.

2. Materials Required

- 2.1 Cut a 3½ foot length of S.C. wire to be tested.  
Note: Do not bend wire unnecessarily.

3. Test Equipment

- 3.1 Springback Test Fixture or equivalent (Fig. 2).  
3.2 5 pound weight.

4. Applicable Documents

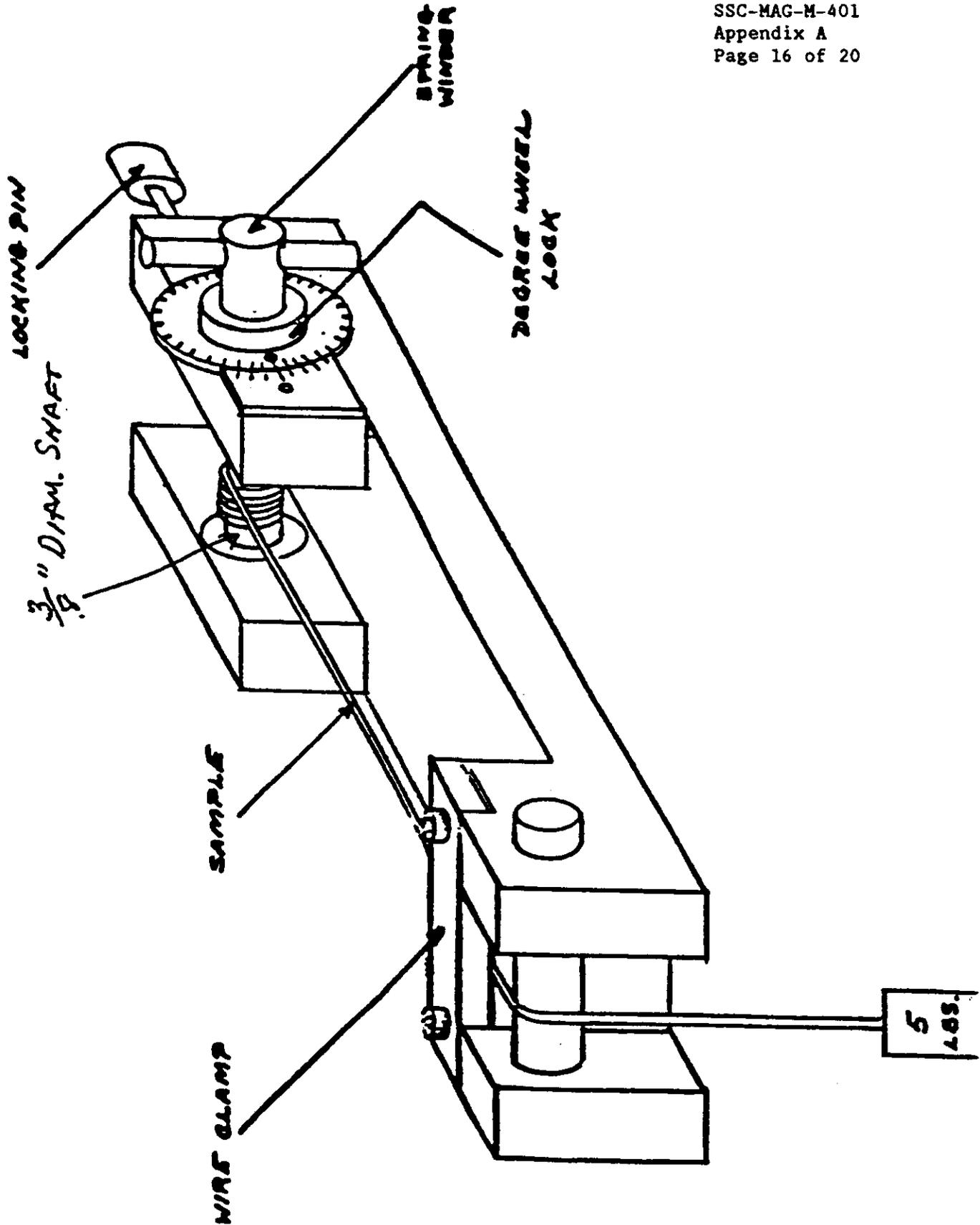
None.

5. Test Procedure

- 5.1 Prepare one end of wire sample with a 1/2 inch 90° bend, and tie the other end securely to a 5 pound weight.
- 5.2 Test the spring fixture to be sure it turns freely.
- 5.3 Thread the 90° bend through the test fixture and place in the hole in the spring winder with the locking pin in place.
- 5.4 Tighten the wire.
- 5.5 Make sure the 90° bend is not affecting the "0" reading and the wire is tangent to the spring winding shaft.
- 5.6 Set "0" on the degree wheel.
- 5.7 Hang the 5 pound weight over the end of the table. Release the clamp. Hold the spring winder handle and pull the locking pin.
- 5.8 Wind 10 complete turns and replace locking pin. Then tighten wire clamp.
- 5.9 Hold spring handle and remove locking pin. Gently let the spring unwind and note the number of revolutions.

- 5.10 Once the spring has stopped, gently touch the spring handle to make sure the spring has equalized and reached its full springback. Do not unwind the spring.
- 5.11 Note and record the total number of degrees of springback.
- 5.12 Cut the sample at the wire clamp and the 90° bend.
- 5.13 Carefully slide the spring winder out of its bearings and remove the sample.
- 5.14 Measure and record the inside diameter, label the sample and store in archives.
- 5.15 Each sample should be measured 3 times.

# SPRINGBACK TEST FIXTURE



Test Method No. 401-5 - Fig. 1.

Test Method No. 401-6 - Determination of Normal State Resistance of NbTi Superconducting Wire.

1. General Outline; Definition of Residual Resistance Ratio

This method covers the measurement of electrical resistance of NbTi multifilamentary composite wire which is used to make high current superconducting cables. The composite matrix is copper. The resistance per unit length is determined at room temperature (295 K) and just above the transition temperature ( $T_c \sim 9.5$  K). These quantities are designated  $R_{295}$  and  $R_{10}$ , respectively, and are measured with an accuracy of 0.5%. The ratio  $R_{295}^{10}/R_{10}$  is defined to be the residual resistance ratio, RRR.

$R_{295}$  is determined chiefly by the copper matrix. For a given wire diameter it provides a measure of the volume copper-to-superconductor ratio (Cu/SC) of the wire.

$R_{10}$  is determined chiefly by the residual resistance of the copper matrix. The ratio RRR provides a measure of the electronic purity of the copper matrix.

2. Apparatus Description

A four wire method is used to determine the resistance. The wire sample is mounted on a probe which is also used for superconducting critical current measurements. It has leads which are suitable for carrying the required current from room temperature into a liquid helium bath, and potential leads for measuring the voltage drop across a measured length of the test specimen. The probe should be mounted so that the test specimen can conveniently be raised and lowered through the level of a helium bath.

Voltage drops are measured with a digital voltmeter of 0.5  $\mu$ V resolution. It is helpful during the low temperature measurement to use an X-Y recorder simultaneously with the digital voltmeter, with Y set to voltage and X to time (see Section 4 below).

Current in the range 0.1 to 1.0 A is provided by a well regulated and filtered DC power supply. It is measured by a shunt of 0.5% accuracy.

In the room temperature measurement a thermocouple device of 0.1° C accuracy is used to determine the ambient temperature.

3. Sample Mounting

The test specimen is wound on a grooved form. The ends are soldered to the copper terminations of the current leads over a minimum length of 1". Voltage taps are soldered to the specimen at a distance of at least 1" from the current joint. Voltage taps are soldered to the specimen at a separation distance of at least 1" from each current lead connection. It is advisable that these taps be in the form of fixed pins so that the test length be

constant throughout a series of measurements. In order to assure an accuracy of 0.2% this length should be 50 cm or more. The voltage leads should follow the sample in a non-inductive fashion so as to minimize noise pickup. Alternatively, the sample may be wound non-inductively on the form.

#### 4. Procedure

Room temperature measurements are made at currents which are a compromise between the requirements of sensitivity and negligible ohmic heating. A typical value is 0.5 A. Voltage readings are taken for forward and reversed current and averaged.

Low temperature measurements are made in a helium dewar. The probe is raised so that the lowest point of the specimen is a few centimeters above the liquid helium bath level while measuring current is flowing. As the sample warms, the voltmeter reading will go suddenly from zero to a finite value corresponding to its normal state resistance. The latter is substantially independent of temperature from the transition temperature,  $T_c$ , to 15 K, so that the voltage remains constant long enough to be read and is recorded. With a reasonably designed probe and former it may take 1 or 2 seconds for the specimen to go normal. The resistance will remain in the residual resistance region several times longer than this. When the X-Y recorder is used, a series of abrupt voltage changes are recorded as the specimen is alternately raised and lowered through the helium bath level. The height of these steps should be reproducible.

#### 5. Room Temperature Correction

Normally occurring room temperature variations may produce significant variations in the measured resistance. Designating this resistance as  $R_m$  and the ambient temperature as  $T_m(^{\circ}\text{C})$ , the resistance at the reference temperature of 295 K is calculated as follows:

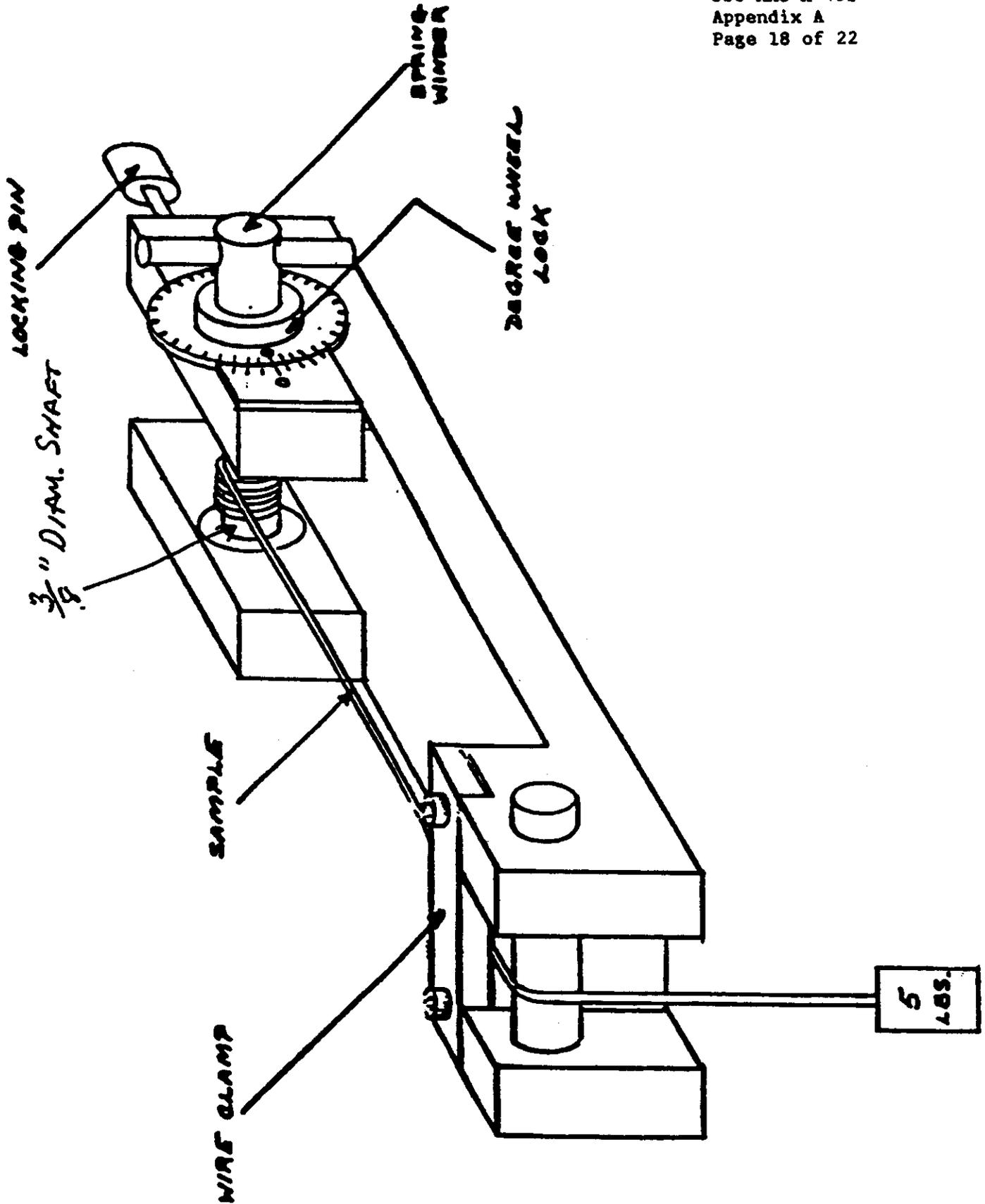
$$R_{295} = R_m / [1 + .0039 (T_m - 22)]$$

The effect of the NbTi is neglected for the purpose of this correction.





# SPRINGBACK TEST FIXTURE



Test Method No. 401-5 - Fig. 1.

