# Superconducting Joints with Rectangular Cross Section Niobium-Tin Multifilamentary Superconductor

by

James Leslie Ludlam

Submitted to the Department of Mechanical Engineering
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## Abstract

Superconducting magnets used in nuclear magnetic resonance (NMR) require high temporal stability of the magnetic flux density. Therefore, they are operated in persistent mode. To maintain a persistent current in the magnet windings, a continuous superconducting path must be available. The common method of constructing NMR magnets utilizes different conductors in the windings, each optimized for its local magnetic field conditions. Since these conductors must be joined together, superconducting joints are required, each one a critical part of the magnet.

This thesis presents the development of a novel approach for the construction of superconducting joints with $Nb_3Sn$ multifilamentary conductor. The method was developed as part of a project to build a 17.6 T (750 MHz) NMR magnet at the Francis Bitter National Magnet Laboratory (FBNML). The $Nb_3Sn$ sections of the magnet use a bronze-process conductor with rectangular cross section, niobium diffusion barrier, and external copper stabilizer. The joints in the $Nb_3Sn$ sections of the 17.6 T magnet are formed by clamping an unreacted composite of compacted niobium and tin powders over exposed filaments at the end of the unreacted conductor. Subsequent heat treatment of the magnet causes superconducting joints to form between the conductor and composite. The joints on the ends of the conductors in each $Nb_3Sn$ section are then connected with NbTi conductors, using a known method, so that the sections are part of a continuous superconducting circuit. The $Nb_3Sn$-to-composite joints produced using the method developed in this thesis project have been able to meet a performance criterion of carrying at least 300 A in a background field of 3 T with a voltage across the joint lower than 7 nV. Some joints were next subjected to a reliability test where they must maintain their performance after being thermal cycled 20 times between room temperature and 77 K. Thermal cycling had no measurable effect on the joints.

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Lastly, special thanks to my wife Kate for her loving support and Gary for his companionship and cerebral conversations. Without these two people, as well as my parents and parents inlaw, none of this would have been possible.

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# Contents

# List of Figures

# List of Tables

# Chapter 1

# Introduction

This introduction discusses superconducting magnets and the types of joints used in them. Superconducting materials are well known to conduct electrons with zero resistance. Of many materials which exhibit superconductivity, only two, NbTi and $Nb_3Sn$, currently find wide spread use in superconducting magnets. The idea of using superconductors to build magnets was put forth almost simultaneously with the discovery of superconductivity by H. Kammerling Onnes. Many uses for superconducting magnets have been envisioned and while most are still only research and development projects, some superconducting magnets are currently used in commercial devices. Joints are frequently required in superconducting magnets to connect different pieces of conductor and can be the determining factor of a magnets performance. There are two main classifications of joints, low-loss and superconducting, the latter being more difficult to build and essential to persistent-mode superconducting magnets.

## 1.1 Superconducting Materials

H. Kammerling Onnes discovered superconductivity in 1911 when he was measuring the resistance of mercury at very low temperatures. He was amazed to find the complete disappearance of the resistivity of the metal at temperatures near 4.2 K. Onnes had been the first to liquefy helium and use it for low temperature experiments. Further experiments by many investigators uncovered superconductivity in other different materials.

The first set of superconducting materials, discovered immediately after the Onnes discovery, later classified as type I superconductors, exhibited the Meissner effect. The Meissner effect is the total exclusion of magnetic field from the interior of the superconducting materials volume. When an external field is applied to a type I superconductor, currents are induced at its surface, shielding the interior of the superconductor from the field. The shielding currents collapse at a critical field and the material loses its superconductivity. The low values of critical field for type I superconductors make them unsuitable for use as magnet conductors. Type II superconducting materials, first discovered in the 1930's, are much more suitable for use as magnet conductors because they can remain superconducting even in very high fields while carrying very high current densities [1].

The three properties which define the limits of a materials superconductivity are critical temperature, field, and current density. When these three properties are measured for a material and plotted in cartesian coordinates they form a surface which covers the origin, intercepting each axis at the point corresponding to the critical value of that axes parameter. Any point between the critical surface and the origin defines a state where the material will exhibit superconductivity. However, even though the critical temperature of NbTi is 9.3 K, for example, it would be of no use to operate a superconducting device at this temperature because the NbTi could not carry any current as the critical current density is nearly zero. Most frequently, the interesting properties are the critical field and current density at 4.2 K because devices are often operated in liquid helium boiling at atmospheric pressure.

When superconducting materials are considered for magnet design there are certain minimum performance criteria which exclude the vast majority of superconducting materials. These criteria are:

1. Critical Temperature $> 10$ K;

2. Critical Field $> 5$ T;

3. Critical Current Density $> 10^9$ A/m$^2$;

4. High Ductility.

On the order of 10,000 materials have been found to exhibit superconducting behavior, but after the criteria shown above are applied only, two materials emerge as useful for constructing superconducting magnets, NbTi and $Nb_3Sn$ [2]. There are other superconducting materials which are potentially useful in high field superconducting magnets. The best materials showing some promise for use in the near future are shown in figure 1-1 [3]. The material $V_3Ga$ is currently available, but only in very small quantities, and $Nb_3Al$ is currently being developed.

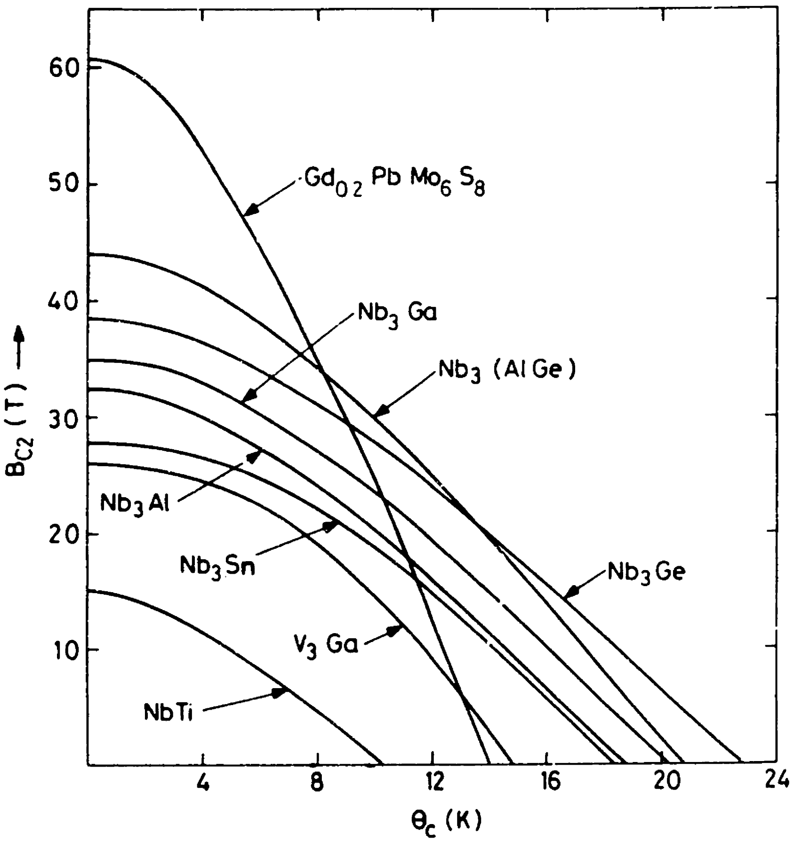


Figure 1-1: High field superconducting materials [3].

The ability of superconductors to pass large currents without generating any joule heating is very useful in magnet design. This fact was identified very early in the development of superconductors. Although early attempts to build superconducting magnets were not successful new, techniques and materials eventually made it possible to construct these devices.

## 1.2 Superconducting Magnets

When Kammerling Onnes discovered the superconductivity of lead and tin in 1913 he noted that these metals, being ductile and easily fashioned into long wires, might be used for "... producing intense magnetic fields with the aid of coils without iron cores" [4]. The first scientists attempting to build such coils were disappointed to find that the superconducting wires they used would only pass a small fraction of the short-sample critical current after the conductor was wound into a coil. These disappointments did not disuade scientists from continuing to experiment with superconducting magnets. Although, complete success would not be realized until the 1960's when type II superconductors were fabricated into high-field, high-current conductors.

While conventional magnets can produce very high magnetic fields, they consume copious amounts of power in the process. The power consumption of a superconducting magnet is always taken as the amount of power required by the refrigeration equipment at room temperature to overcome the refrigeration load generated by the magnet during operation. While the amount of generated heat can be very small, the large temperature difference between the magnets operating temperature (most often that of liquid helium boiling at atmospheric pressure, 4.2 K) and the efficiency of the refrigeration equipment means that the room temperature power input must be many times greater, often 500 to 1000 times. For example, the Big European Bubble Chamber at CERN consists, in part, of a large superconducting solenoid producing 3.5 T in a 4.7 m bore. The magnet system generates a refrigeration load of 900 W in the cryostat corresponding to a room temperature input of 360 kW to the refrigeration equipment. In contrast, a water cooled copper magnet would consume around 60 MW [3]. It is clear from this example that the operating cost for superconducting magnets are much lower than for equivalent conventional magnets. In addition, for large systems, the capital costs associated with a superconducting magnet and the accompanying equipment can be less than that for a conventional magnet system.

There are several areas where superconducting magnets are useful or potentially useful. Areas where superconducting magnets are currently used include physics research such as particle accelerators and magnetically confined plasma fusion reactors. Commercial devices with superconducting magnets include research magnets for producing high magnetic flux densities in the laboratory, nuclear magnetic resonance (NMR) spectrometers for sample analysis, and magnetic resonance imaging (MRI) for medical diagnosis. There are several other potential uses for superconducting magnets including magnetically levitated trains, magneto-hydrodynamic propulsion and power co-generation, superconducting magnetic energy storage (SMES), and superconducting motors and generators. While all of the potential uses are in the research and development phase, prototype devices have been built to demonstrate the feasibility of many of these ideas. Many of them could be commercially viable with some changes in the economic environment.

Magnets used for NMR and MRI are of particular interest to this project because they must produce magnetic flux density of very high spatial homogeneity and temporal stability. To achieve the high temporal stability the current flowing in the magnet windings must remain constant by operating the magnet in persistent mode or with a supply of extremely high stability. Persistent-mode magnets are currently much more popular than driven-mode magnets because of the difficulty in producing a current source of high enough stability. If the magnet is operated in persistent mode then any voltage generated in the magnet windings will cause the current to decay, ruining the temporal stability. The most frequent cause of undesirable voltage is resistance in joints between conductors in the magnet windings. These joints are critical parts of an NMR or MRI magnet and they are often the determining factor of the magnets performance.

## 1.3 Joints

Joints are used to connect all of a magnets windings in order when a single length of conductor is not long enough to build a winding and must be joined with other lengths of conductor. They can also be used between separate sections of a magnet to allow different conductors to be used at different places in the magnet as the magnetic field and internal stresses change. This allows the specifications of each conductor in a magnet system to be tailored to the local conditions which it will experience during operation. Some joints connect two conductors of the same type and some are hybrid joints, connecting different types of superconductor. The superconductors used in modern magnets have many small superconducting filaments embedded in a matrix of normal metal (multifilamentary conductors) which adds to the complexity of building joints.

There are two basic types of joints which are used in superconducting magnets, low-loss and superconducting. Low-loss joints have some nominal resistance resulting in a heat load imposed on the cryostat system. Low-loss joints are not suitable for use in persistent-mode magnets but they can be used in driven-mode magnets. Superconducting joints are truly superconducting and can be used in magnets which continuously carry current without the aid of a supply (persistent mode). Superconducting joints are usually placed in low-field regions within the cryostat so they can carry as much current as possible. This is necessary because the joining process can reduce the critical properties of the joint to values less than those of the conductor.

### 1.3.1 Low-Loss Joints

Low-loss joints have a small nominal resistance due to the presence of normal metal interposed with conductors which are being joined. Current must flow through this material when passing from one conductor to the other. A common low-loss joint is a lap joint where two conductors are soldered together as shown in figure 1-2. Current flowing from one conductor to the other must pass through the normal metal, generating joule heating. Although some solders are superconducting, they have very low critical field and are generally resistive when carrying current in a magnetic field. The resistance and resulting refrigeration load imposed on the cryostat system can be calculated for each joint in a magnet [3]. The small resistance in the joints requires that a current supply be used to maintain the current during continuous operation.

Since low-loss joints introduce a small resistance to the magnet windings, they

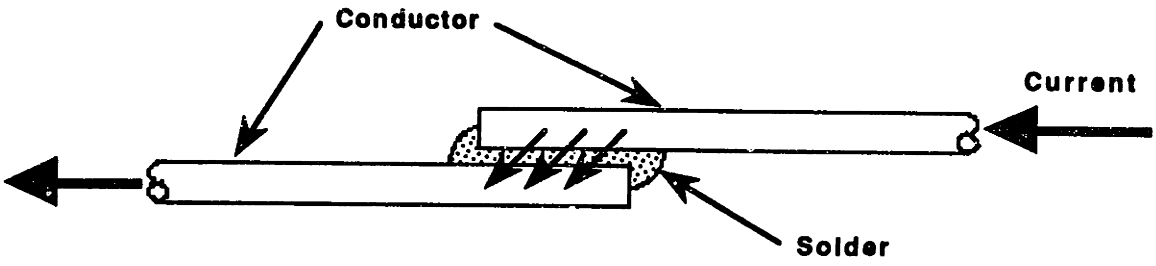


Figure 1-2: Low loss joint.

cannot be used in magnets intended for persistent-mode operation. The resistance would cause the field to decay over time without the impetus of the supply. Magnets used for NMR and MRI must have very high temporal stability in the fields they produce, decay rates on the order of one part in $10^8$ or less are generally required. Therefore, joints used in persistent-mode magnets must be superconducting.

### 1.3.2 Superconducting Joints

In a persistent-mode magnet, the power supply is only used to charge the magnet to a desired operating current and to discharge the magnet when necessary. For persistent-mode operation the magnet terminals are shorted together with a superconducting switch. Under ideal conditions the current would persist and flow indefinitely, however there is a very small nominal voltage in the magnet circuit which causes a very slow current decay thus decreasing the field over time. Frequently, resistance in the 'superconducting' joints causes the field decay. This means that the quality of the joints is critical in persistent-mode magnets. The operation of a persistent-mode magnet is shown in figure 1-3.

The current and voltage in a persistent-mode magnet are related by the simple equation

$$V = L\frac{dI}{dt} \tag{1.1}$$

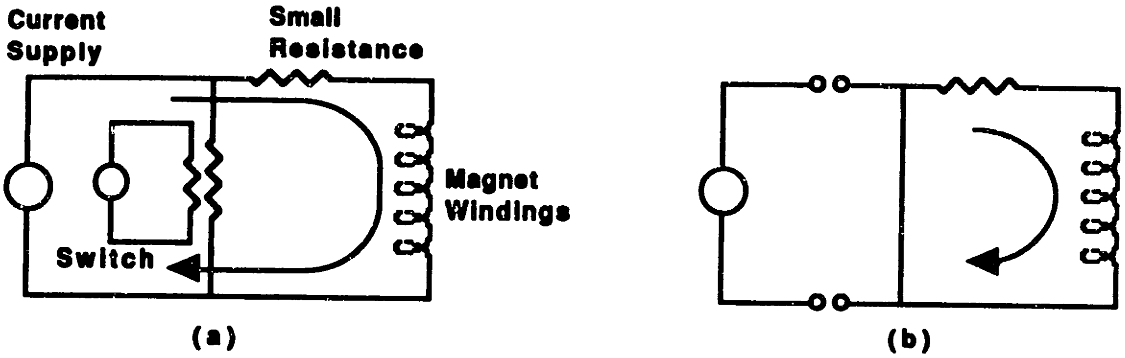


Figure 1-3: (a) Persistent mode magnet being energized. (b) Magnet operating in persistent mode.

which can be written as

$$\frac{1}{I}\frac{dI}{dt} = \frac{V}{IL} \tag{1.2}$$

A typical temporal stability requirement for NMR magnets is 0.01 ppm (parts per million), thus $V/IL = 10^{-8}$ per hour. The total voltage in the circuit consists of the voltage drop in all the coils plus the voltage drop for N joints in the circuit.

$$V = V_{coil} + \sum_{n=1}^{N} V_{jn} \tag{1.3}$$

Usually $V_{coil}$ can be considered negligible compared with the total of the joint voltages and the performance criterion can be written

$$\frac{\sum_{n=1}^{N} V_{jn}}{IL} = 10^{-8}/\text{hr} \tag{1.4}$$

### Thesis Objective

The objective of this thesis work is to develop superconducting joints for use in the $Nb_3Sn$ sections of a 17.6 T (750 MHz) NMR spectrometer magnet system currently being developed at the Francis Bitter National Magnet Laboratory (FBNML). The joints are required in all of the $Nb_3Sn$ sections of the magnet and they must be connected together with NbTi conductors similar to the method used in a 14.2 T NMR magnet built at FBNML [5]. The 17.6 T magnet consists of 12 separate coils arranged as shown in figure 1-4 and will be used at frequencies up to 750 MHz. The dimensions and peak field values are given in table 1.2 where:

$a_1 \equiv$ winding inner radius;
$a_2 \equiv$ winding outer radius;
$z_{med} \equiv$ axial displacement of coil center from magnet center;
$2b \equiv$ axial length of coil;
$B_{pk} \equiv$ peak field.

The seven innermost sections are made using $Nb_3Sn$ conductors while the outer sections are exposed to lower peak fields and are wound with NbTi conductors. This arrangement requires a total of at least 14 $Nb_3Sn$ joints and at least 10 NbTi joints. An important difference between the joints for the 17.6 T magnet and those for the 14.2 T magnet is that the conductor being used in the former has rectangular cross section while the conductor in the latter had round cross section.

Reliable methods are available for producing the joints in multifilamentary NbTi conductor so it is assumed that the voltage generated in the coils will be dominated by the $Nb_3Sn$ joints. This leaves the 14 or so $Nb_3Sn$ joints as potential sources of voltage. Assuming that I=305 A, L=110 H, and $n_j=14$ we can calculate the maximum permissible average voltage in each joint.

$$V_j = \frac{(10^{-8}/\mathrm{hr})(1\mathrm{hr}/3600\mathrm{s})(305\mathrm{A})(110\mathrm{H})}{14} = 6.7\mathrm{nV} \qquad (1.5)$$

Therefore each joint, on average, must not produce a voltage drop of more than 6.7 nV while the magnet carries its operating current of 305 A (*e.g.* its effective resistance should be less than 22 pΩ). Since the magnetic field from the magnet itself affects the current carrying capacity of the joints, they must be situated some distance from the magnet in the cryostat so that they experience a peak field of less than 1.5 T. Therefore, a conservative minimum performance criterion for each joint requires that it carry 300 A in 3 T field with less than 7 nV. This criterion was imposed on each joint for the 17.6 T NMR system.

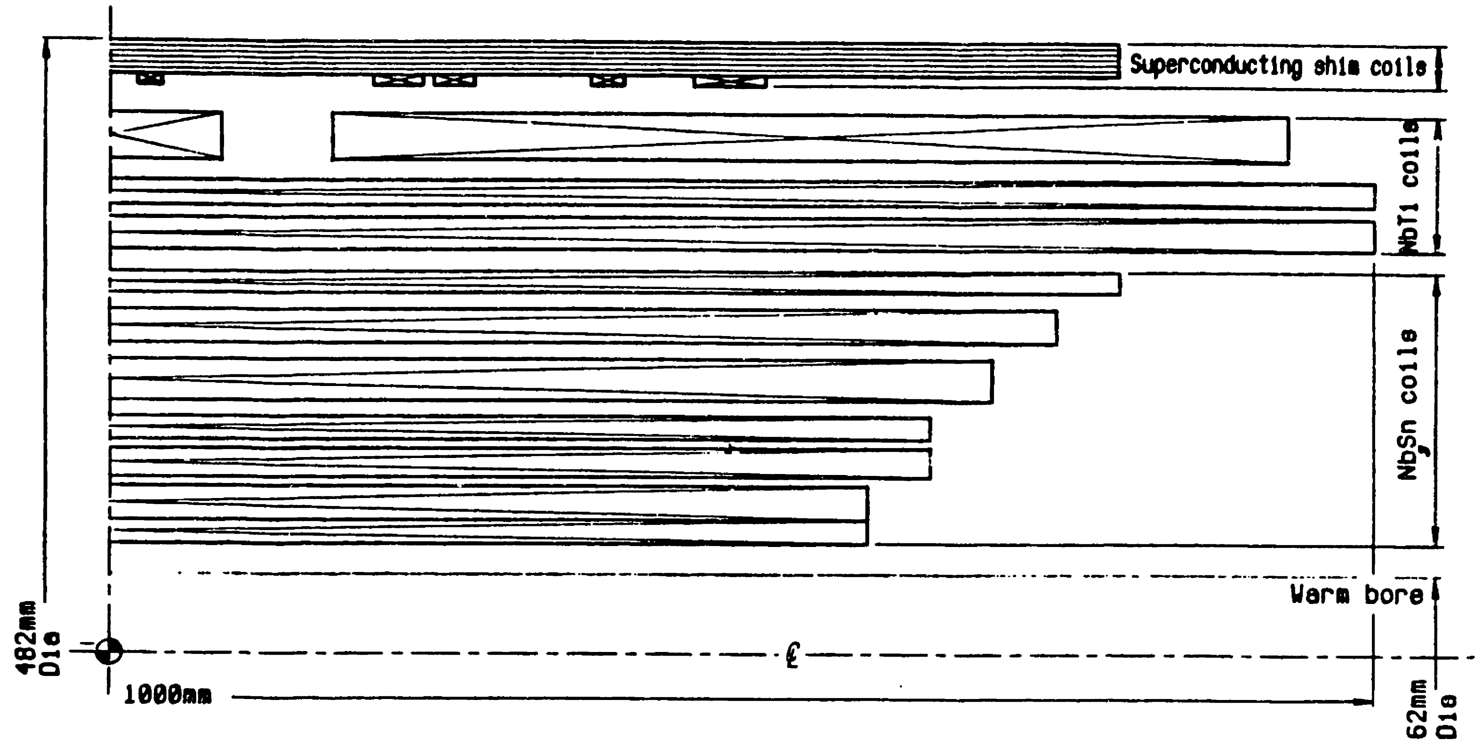


Figure 1-4: 750 MHz NMR Magnet Schematic

| | $Nb_3Sn$ Coils | | | | | | | NbTi Coils | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Coil 1 | Coil 2 | Coil 3 | Coil 4 | Coil 5 | Coil 6 | Coil 7 | Coil 8 | Coil 9 | Coil 10 | Coil 11 | Coil 12 |
| $a_1$ (mm) | 43.0 | 52.1 | 68.8 | 83.4 | 99.0 | 121.7 | 141.3 | 158.0 | 176.0 | 194.2 | 194.2 | 194.2 |
| $a_2$ (mm) | 52.1 | 65.3 | 79.9 | 92.5 | 115.2 | 134.8 | 149.5 | 171.0 | 186.2 | 212.5 | 212.5 | 212.5 |
| $z_{med}$ (mm) | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 280.1 | −280.1 |
| 2b (mm) | 600 | 600 | 650 | 650 | 700 | 750 | 800 | 1000 | 1000 | 88.7 | 377.1 | 377.1 |
| $B_{pk}$ (T) | 17.6 | 17.0 | 16.0 | 15.0 | 14.0 | 11.9 | 9.9 | 8.5 | 6.1 | 4.7 | 4.7 | 4.7 |

Table 1.1: 750 MHz NMR magnet specifications

# Chapter 2

# Background and Discussion

This chapter discusses the development of superconducting magnets and joints. The section on early magnets and joints covers the period from 1911 to 1969 and the section on modern magnets and joints covers from 1969 to the present. In each section examples of joints typically used in the magnets are given. Some of the joint building methods were reported in the literature and other examples are taken from patents. Joint building methods involving $Nb_3Sn$ conductors have always been most difficult. In the case of the 750 MHz NMR magnet, additional difficulties were presented by the decision to use conductors with rectangular rather than round cross section.

## 2.1 Early Magnets and Joints

Early superconducting magnets were built to see if such devices were feasible. Some coils were powered by an external supply and some were built to explore persistent mode operation. All of these coils were made with monofilament superconducting wire. The experiments by Onnes spawned a great deal of curiosity about superconducting magnets and other researchers began building test coils. Each experimenter would find his own favorite technique for producing joints in the magnet and between the magnet and its current supply. Joints in the coil winding were often avoided and low-loss joints connecting the coil and supply became the focus of joint making. The methods, developed in the 1950's, usually consisted of clamping the two conductors together or fusing them together with welding or heating [6, 7, 8]. These types of joints continued to be popular after the discovery of type II superconductors because they worked well with the ductile superconducting alloys NbZr and NbTi. After 1965 when NbTi was discovered to be an excellent superconductor it quickly became the most popular material in superconducting magnets because of its high ductility, critical field, and current density.

The joints produced in NbZr and NbTi conductors were generally made by forcing the conductors together with spot welding or clamping [9, 10]. The contact caused by forcing the conductors together was sufficient to create superconducting paths in the finished joint. The excellent superconducting properties of $Nb_3Sn$ were known, but the brittle nature of the material made it very difficult to form joints using these methods. Some experimental coils were built with $Nb_3Sn$ conductor but joints were avoided except for low-loss connections to the current leads [11].

These types of methods for constructing joints in the ductile monofilament superconductors persisted throughout the 1960's. Typical examples of these types of joints are shown in US patents 3,422,529 and 3,527,876 (Appendix A). In the first patent, the joint is made by twisting two NbZr monofilaments together and placing them inside a hollow piece of the same material. The piece is then crimped to force the filaments together. This is an example of using clamping force to make a joint. In the second patent the joint is made by spot welding two NbZr monofilaments to a NbTi plate. This method claimed to be superior to spot welding two conductors together directly.

During the 1960's it began to become clear why coils wound from single filaments of superconducting wire would not perform as well as originally expected. The discovery of flux jumping led to the conclusion that stable magnets must have superconducting filaments smaller than a certain critical size and that some normal metal was required along with the superconducting material. Following this, conductors consisting of many small filaments (typically less than 100 $\mu$m dia.) in a matrix of normal metal were built and used to construct magnets. These multifilamentary conductors became the standard material for magnet construction and magnet designers finally achieved the goal of reaching short sample critical current values in an actual magnet.

## 2.2 Modern Magnets and Joints

In this section the theory of flux jumping and how it relates to the development of multifilamentary conductors is briefly introduced. Following that, examples of modern superconducting magnets and their joints are given. Since this work is part of the project to build the 17.6 T NMR magnet, examples concentrate on superconducting joints in persistent-mode magnets. In particular, the method of Williams [5] is discussed in detail because it formed the foundation for the method developed in this thesis.

The emphasis in these examples in on superconducting joints for use in persistent mode magnets since that is directly related to the 17.6 T NMR project which motivated this work.

### 2.2.1 Flux Jumping

The understanding of the phenomenon of flux jumping in the 1960's was critical to the development of modern superconducting magnets. Flux jumping is a thermal instability phenomenon initiated by heat disturbances. Assume that a superconductor in a magnet with current flowing through it is exposed to a small heat disturbance. This disturbance creates heating in the superconductor which raises its temperature and lowers its critical current density. The decrease in the critical current density forces the screening currents in the superconductor to redistribute themselves allowing the magnetic flux to further penetrate the superconductor. The motion of the flux causes a small amount of heating to occur, raising the temperature of the superconductor further and compounding the problem. The resulting runaway temperature increase is called a flux jump and will result in the magnet conductor loosing its superconducting properties and returning to the normal state (quenching). The figure 2-1 shows diagrammatically how this process works.

The theory of flux jumping is covered in detail in the literature [3, 1]. The theory

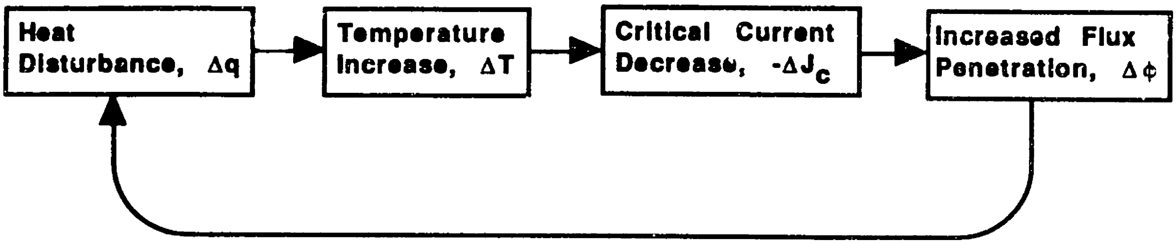


Figure 2-1: Process of flux jumping.

leads to the conclusion that there is a maximum critical dimension for the superconducting wires used in a magnet. Since this dimension is typically much less than 100 $\mu$m it is not practical to wind magnets from single filaments. As a result, conductors were fabricated with many filaments mechanically and electrically bound with a normal metal matrix, usually copper. These multifilamentary conductors allow many superconducting filaments to be wound into a coil very easily.

In multifilamentary conductors were developed. In these conductors, many filaments are mechanically and electrically bound inside of a normal metal matrix.

One problem with early multifilamentary conductors was that the metal matrix in which the filaments were embedded allowed the filaments to be coupled under the influence of external fields and the current flowing in the filaments. The coupling due to external fields can be made negligible by twisting the filaments thereby producing a net cancellation of the external field effects. The self field effect remains but can be made insignificant by using a matrix material which has high thermal diffusivity and low magnetic diffusivity, such as copper. If the characteristic time for heat diffusion in the conductor is much greater than that for magnetic flux diffusion then the heat can be conducted away by the matrix metal before the flux jump can proceed. In this case the external cooling around the perimeter of the conductor becomes important.

### 2.2.2 Multifilamentary Conductors

Multifilamentary conductors are standard for use in modern superconducting magnet windings. Figure 2-2 shows the cross sections of two examples of multifilamentary conductors. The magnified views in the figure show the filaments embedded in the metal matrix. The results of research during the 1960's were compiled in a paper

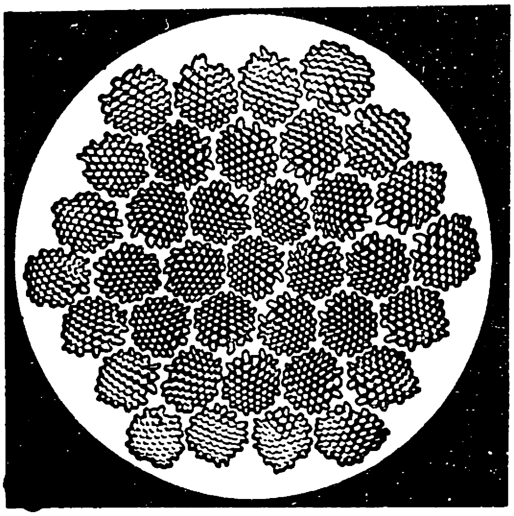


Figure 2-2: Example of multifilamentary superconductor about 1mm in diameter. The smallest structures visible are filaments in the matrix [3].

written in 1969 by the Superconducting Applications Group at Rutherford Laboratories [12]. This paper served as the starting point for many magnet designers in the 1970's as they began constructing various high performance superconducting magnets. Many practical issues concerning the design of practical conductors and magnets are covered in this paper.

The preeminant superconductors today are multifilamentary composites using $Nb_3Sn$ or NbTi. Other conductors such as $Nb_3Al$ and $V_3Ga$ appear promising for future use but they are still being developed. All multifilamentary conductors are produced in similar ways. Large billets of the bulk materials are hot extruded to reduce their diameter, forming smaller rods. The smaller rods are then bundled together and extruded again. This process is repeated until the desired number and size of filaments is reached. The final rod containing all the filaments is then drawn through dies to reach the desired dimensions for the conductor. The process for NbTi is shown in figure 2-3 as an example of multifilamentary conductor fabrication [3]. The process is similar for $Nb_3Sn$ but the Nb and Sn are contained in separate components during fabrication. The $Nb_3Sn$ forms after a heat treatment where the tin diffuses into the niobium. Several different ways of building $Nb_3Sn$ multifilamentary composite con-

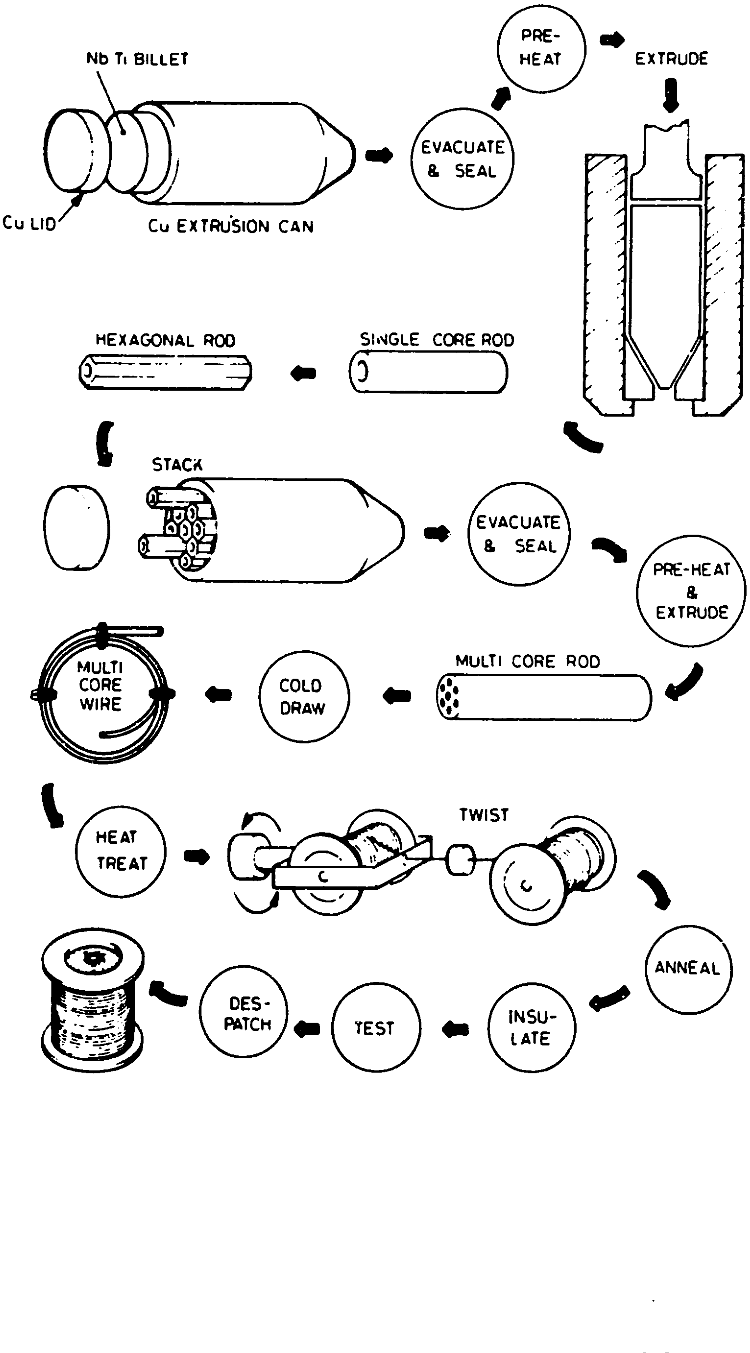


Figure 2-3: Fabrication process for NbTi [3].

ductors are shown in figure 2-4. In each case copper stabilizer would be added later along with a diffusion barrier to keep the stabilizer from being contaminated during heat treatment.

The increased complexity of the multifilamentary conductors has made it more difficult to fabricate the joints in superconducting magnets. Some clamping and welding techniques are still used for NbTi because of its ductility. Methods for joining $Nb_3Sn$ are much more difficult due to the fragile nature of the conductor after reaction.

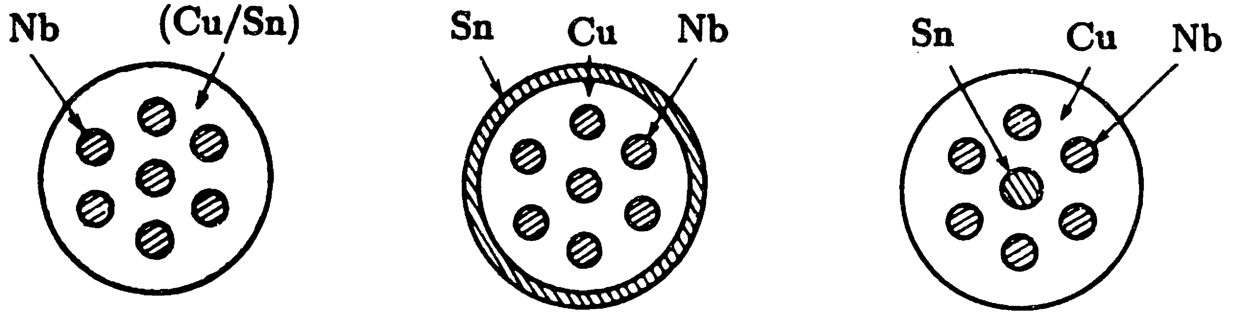
### 2.2.3 Magnets and Joints

#### NbTi

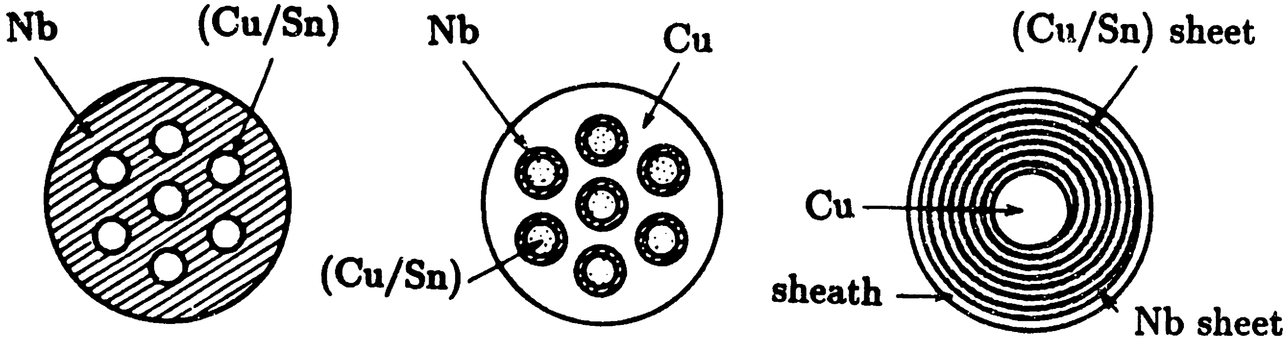
Superconducting joints for use in persistent mode magnets are relatively easy to build with NbTi conductors. Since it is ductile the filaments can be compressed and forced into contact with each other. Joints for a 6.5 T NMR magnet were made by pulse welding NbTi filaments to NbTi foil. The filaments were freed from the matrix and arranged on the foil then the filaments and foil were welded together [13]. Another widely used method to form the joint is to expose the filaments by etching away the matrix metal with acid and then crimp the filaments in a sleeve. In a 7 T NMR magnet built at the Francis Bitter National Magnet Laboratory (FBNML), the joints were formed by crimping the exposed filaments in a copper sleeve [14]. It is also possible to use a superconducting solder such as lead bismuth eutectic to join two conductors filaments. Examples of these joints are shown in patents 4,713,878 and 4,901,429 in Appendix A. In both of these examples the matrix material is removed by a bath of molten metal. Then, the filaments are placed in a mold with the molten solder and the mold is allowed to cool.

#### $Nb_3Sn$

In a 14.2 T NMR magnet built at FBNML, joints were required in the $Nb_3Sn$ sections of the coil [5]. The joints were produced using round $Nb_3Sn$ conductor. They are located in a magnet area where the field is lower than 2 T. A novel technique was



Bronze External Diffusion Internal Diffusion



Solid Solution Reaction Niobium Tube Jelly-Roll

Figure 2-4: Fabrication methods for $Nb_3Sn$ conductors.

developed to make these joints: a $Nb_3Sn$ conductor (circular cross section) from each winding is joined to 4 NbTi monofilament conductors via a powder based $Nb_3Sn$ composite post. A schematic of the joints is shown in figure 2-5.

The first step in building these joints is to force the unreacted conductor from a winding into a hole drilled in the unreacted composite post. [1] A short length of exposed filaments is butted against the bottom of the hole. The conductor is forced against the bottom of the hole, using a modified cold welder, until the conductor expands in the hole. Inserted this way, the conductor is gripped quite firmly by the composite. After heat treatment, the end of the conductor is bonded to the composite in the butt area, forming a superconducting joint. Both ends of the conductor in each $Nb_3Sn$ winding are terminated in this fashion.

After heat treatment, the reacted windings containing the $Nb_3Sn$ conductor to $Nb_3Sn$ composite superconducting joints must be connected together. To do this, four NbTi monofilament conductors are attached to the composite post. The NbTi conductors are attached to an area where the copper sheath of the composite was removed prior to heat treatment. This area is polished after reaction so that the surface is as smooth as possible. One end of each NbTi conductor, a few centimeters in length, is etched to remove the copper and flattened. Each length of the NbTi 'ribbon' is attached to the polished part of the $Nb_3Sn$ composite post. The superconducting connection is made by placing a $Nb_3Sn$ tape between the polished section of the $Nb_3Sn$ composite post and the NbTi ribbon and spot welding the pieces together. The weld extends through the NbTi ribbon, $Nb_3Sn$ tape, and the composite. These plugs of weld material contract more than the surrounding material when the joint cools. The differential thermal contraction causes the three components to be held together very tightly and superconducting paths form across the interfaces between the composite, tape, and ribbon.

The joints in the 14.2 T NMR magnet are called hybrid because they connect two different types of superconductor, $Nb_3Sn$ and NbTi. These joints actually consist of two connections, one between the $Nb_3Sn$ conductor and the $Nb_3Sn$ composite and a

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[1] See section 3.1.1 for a description of the composite and its fabrication

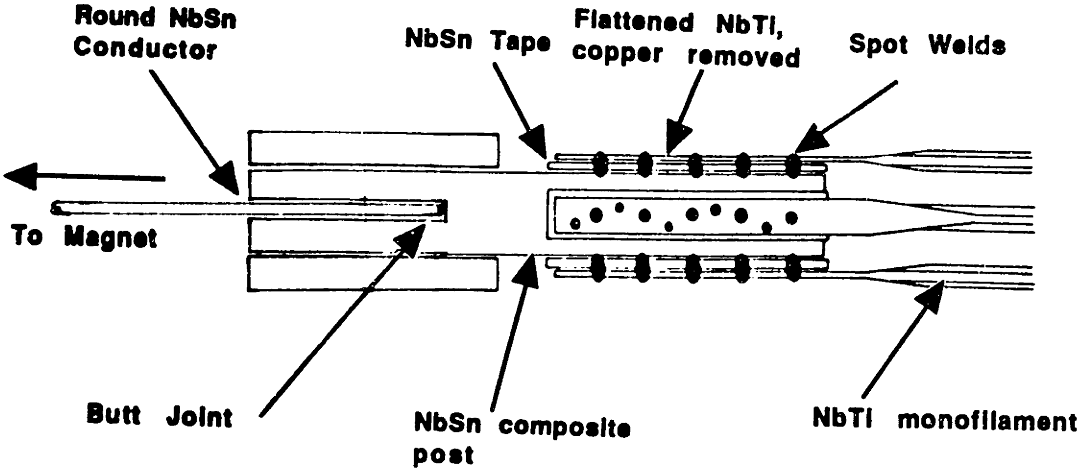

Figure 2-5: 14.2 T NMR magnet joints (adapted from [5]).

second between the composite and the NbTi conductor. In the current 750 MHz, 17.6 T NMR magnet project, the focus of the joint building is on the connection between rectangular cross section $Nb_3Sn$ conductor and the $Nb_3Sn$ composite. The goal is to develop a technique to fabricate reliable connections. Once the $Nb_3Sn$ conductor to composite joint is built then the composite to NbTi conductor connection can be built with the spot welding method described above.

### 17.6 T NMR Magnet Project

In this project superconducting joints capable of passing a current above 300 A in 3 T without exceeding the maximum permissible voltage of 7 nV are required. This is a conservative performance criterion since the joints will be placed in a low field section where the ambient field is not expected to exceed 1.5 T. The joints used in the 14.2 T NMR magnet were used as a model for this work. Since reliable methods were available for producing both NbTi joints and the composite-to-NbTi part of the hybrid joint, this work focused on building the $Nb_3Sn$-to-composite connection using the rectangular cross section conductor actually used in the 17.6 T NMR magnet.

Early in the work on these joints, many different types were tried, attempting to duplicate the success of the joints in the 14.2 T NMR magnet. Success was limited and some new approaches to connecting the $Nb_3Sn$ conductor and composite were explored. The approach which was finally settled on is described in the next chapter on apparatus and procedure for building and testing the joints.

# Chapter 3

# Apparatus and Procedure

This chapter describes the apparatus and procedure used for building and testing the joints developed for the 17.6 T NMR magnet project. The joints are built by clamping composite pieces over exposed filaments on the end of a conductor and heat treating the whole assembly, forming continuous paths of $Nb_3Sn$ in the composite and conductor. After the heat treatment the joints are reinforced with epoxy and soldered into a sample holder for testing. The dimensions of all the joints built are given in Chapter 4 where results are discussed.

## 3.1 Joint Building

### 3.1.1 Apparatus

There are two parts to the joint, the conductor and the composite. The conductor used in these joints is rectangular cross section $Nb_3Sn$ multifilamentary bronze-processed conductor with external copper stabilizer and a niobium diffusion barrier. The diffusion barrier is located between the bronze matrix and the copper stabilizer. The barrier prevents tin from diffusing from the bronze into the stabilizer and contaminating it. Contaminated copper stabilizer has an increased resistivity, making it less effective. The composite starts as a copper tube containing niobium and tin powders. The tube is 25 mm od with a wall thickness of 1.6 mm. The niobium and tin powders have particle sizes of 70–150 $\mu$m and 10-15 $\mu$m respectively. The powders are combined in the stoichiometric ratio and well mixed before they are put into the copper tube. Once the powder is in the tube it is sealed and evacuated. The sealed tube is put through rollers to reduce the area, compact the powder, and form the nearly square cross section of approximately 9 mm. When this material is heat treated, the tin diffuses into the niobium and forms $Nb_3Sn$ . A sketch of the composite and its processing is shown in figure 3-1.

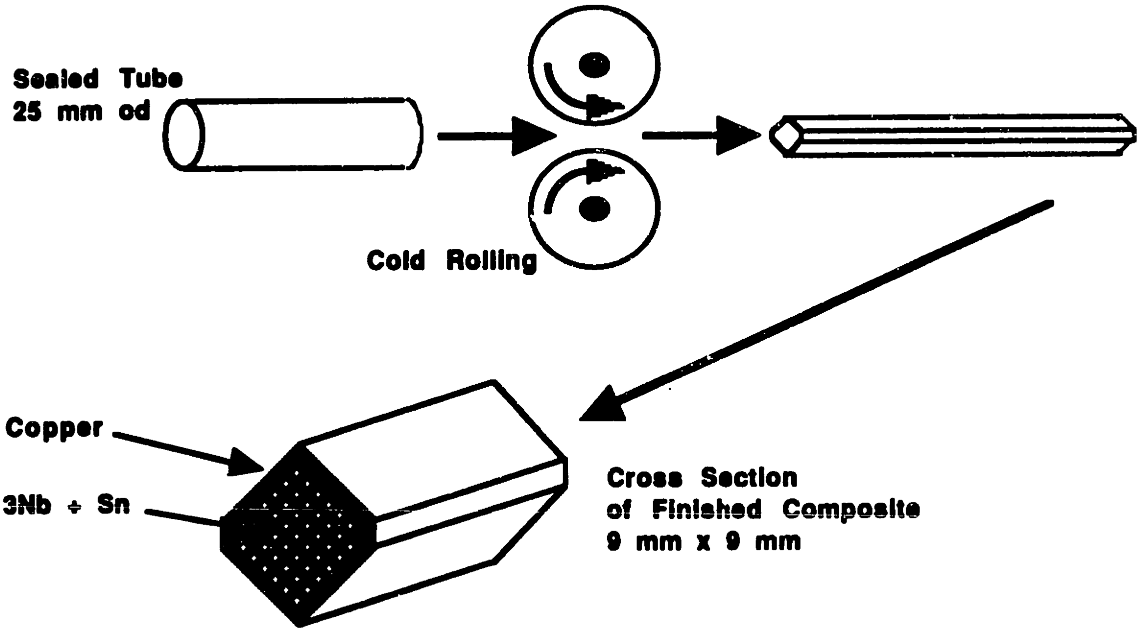

Figure 3-1: Composite material consisting of fine particles of niobium and tin compacted within a copper jacket.

To build the experimental joints, short sections (about 25 mm) are cut from the composite. These are cut into two pieces and clamped over the end of the conductor to form a termination. The composite and conductor are held together during heat treatment so the a superconducting joint between the conductor and composite forms.

### Clamping Fixture

To build these joints, exposed conductor filaments are clamped between the two halves of a composite piece and the whole assembly is heat treated. To accomplish the clamping, the fixture shown in figure 3-2 was built. Heat treatment temperature

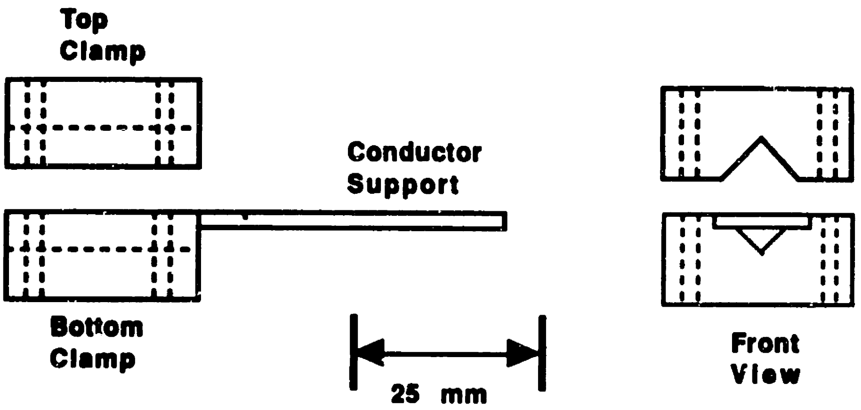

Figure 3-2: Joint Clamping Fixture.

is 700 °C and to avoid reaction with the joint pieces the clamping fixture is built from stainless steel (type 316). The clamp holds the composite pieces diagonally to make it easier to remove the pieces after heat treatment. The composite protrudes slightly from the clamp so that epoxy can be applied to the joint pieces after heat treatment. The joints are very fragile after heat treatment so the epoxy is added to provide some support and reduce the risk of damage during handling. The extension off the side of the clamp provides support for the conductor during heat treatment. Stainless steel wire is wrapped around the extension and conductor to keep the conductor from warping during heat treatment. The clamps are heated in air with a torch to produce an oxide layer which prevents the joint from sticking to the clamp after heat treatment.

## Heat Treatment Equipment

Once the joints are assembled in the clamping fixtures, they are placed into a furnace for heat treatment at 700 °C. The duration of the heat treatment is varied for different joints. Inside the oven is a stainless steel retort which can be evacuated and then filled with helium gas to provide an inert environment for the joints during heat treatment. It is important to ensure that the inside of the retort is free from any grease or other contaminants because small amounts of impurities can react with the joint pieces during the heat treatment. The schematic in figure 3-3 shows the heat treatment furnace and helium supply. The helium is supplied from a compressed gas cylinder.

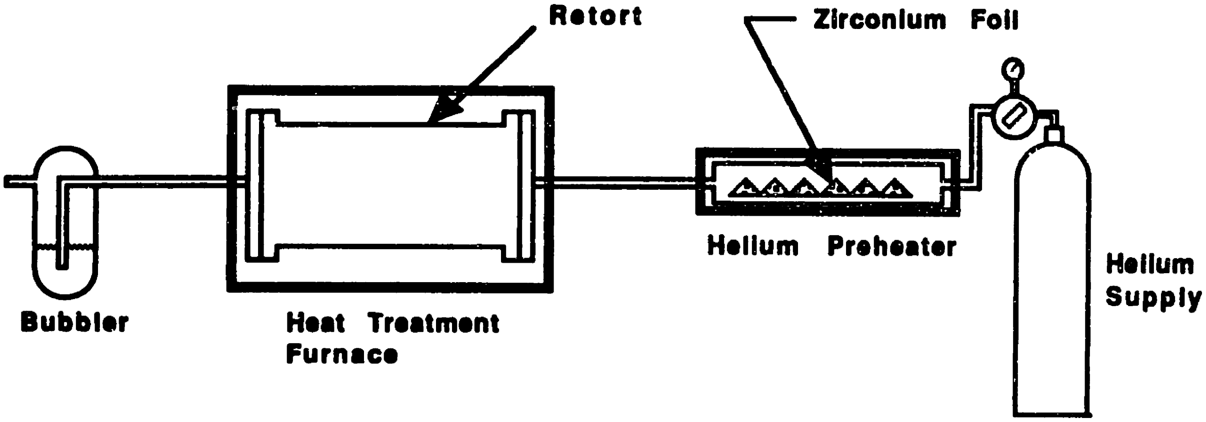

Figure 3-3: Heat treatment furnace and helium supply.

A preheater heats the gas to 700 °C, which in the presence of zirconium foil, removes impurities. The gas then flows into the furnace and the retort, exiting through a bubbler, which prevents the backflow of air.

### 3.1.2 Procedure

The procedure for building joints consists of four parts:

1. Conductor preparation;
2. Composite preparation;
3. Assembly and heat treatment;
4. Final assembly.

Conductor preparation exposes the filaments of the conductor so they can contact the composite. Composite preparation consists of machining the composite pieces so that they will grip the conductor. The conductor is gripped to give it some support during handling. A recessed area is machined in the composite to minimize filament damage. After assembly and heat treatment, the final assembly consists of steps taken to strengthen the joint and prepare it for testing. There are small differences in the procedure details among joints which are discussed in Chapter 4.

### Conductor Preparation

Once a length of conductor for the joint is cut to size, some niobium filaments are exposed so they can come into direct contact with the composite. The three-step etching procedure is used:

1. Remove copper stabilizer with a 1:1 solution of $HNO_3$ and $H_2O$;
2. Remove the niobium diffusion barrier with a 1:1 solution of HF and $HNO_3$;
3. Remove bronze matrix with a 1:1 solution of $HNO_3$ and $H_2O$.

The first step is very easy to accomplish. The copper is etched away in about one or two minutes leaving the diffusion barrier completely exposed. Step 2 is much more difficult because the reaction proceeds quite quickly and very vigorously. Only a few seconds are required to remove the niobium barrier. Leaving the conductor in the solution too long allows the matrix and filaments to be attacked. The etching in step 2 is stopped when the matrix is visible over about 80–90% of the area being etched. This is adequate for step 3 which etches all of the bronze matrix material. The third step proceeds without difficulty and takes a few minutes before the matrix is completely removed. The exact length of filaments exposed varied between joints and is discussed in Chapter 4.

### Composite Preparation

To clamp the conductor and filaments inside the composite, it is necessary first to cut the composite into two halves. During heat treatment, the filaments clamped between the composite halves fuse to the composite and form a superconducting path. Since the area where the joint occurs is structurally very weak, it is positioned some distance inside the composite piece. This requires the composite pieces to be machined to accept a length of the unetched conductor. Care must be exercised to grip the conductor along its entire length. Thermal contraction of the free length during cool down might damage the joint. Figure 4-1 shows the actual dimensions that were used to machine each composite piece.

### Assembly and Heat Treatment

As discussed above, assembling a joint consists of placing the conductor and exposed filaments between the composite pieces and clamping them together. Care is taken to ensure that the filaments are all inside the composite to minimize filament damage. In some cases an extra amount of tin powder is placed inside the joint area for the exposed niobium filaments to react with. While the clamp and joint pieces are being held together the bolts are put into place and tightened as much as possible with a wrench and nut driver. After tightening there is no gap between the two pieces of the composite. Dry graphite lubricant is placed on the clamp groove surfaces and the nut and bolt threads to prevent seizing. It is important not to use any other kind of lubricant as it will introduce impurities which will corrode the joint, clamp, and retort.

The clamped joints are placed in the retort, which is sealed and evacuated. During furnace heating, the retort is periodically flushed with helium and purged to remove contaminants. This procedure is generally repeated four or five times before the temperature inside the retort reaches 200 °C then the helium is allowed to flow at a slow, constant rate. The furnace requires approximately two hours to reach 700 °C from room temperature and about fifteen hours to cool from 700 °C to about 100 °C, at which point the retort can be opened. Exact heat treatment times for the different joints are given in Chapter 4.

### Final Assembly

After the joints are removed from the furnace and before the joints are freed from the clamps, a small amount of epoxy is applied to the area where the conductor protrudes from the composite. The epoxy provides additional strength allowing the joints to be removed from the clamp more easily and without damage. The clamps are disassembled and the joints are removed. This is the stage when the joints are most likely to be damaged and anything which makes it easier to remove the joints from the clamps without damage is beneficial. Once the joints have been removed, additional epoxy is applied along the break in the composite. After this epoxy cures the joints can be handled and prepared for testing.

Some minor work must be performed on the joints before they are ready to be tested. Extra leads must be attached to the end of the joint opposite the conductor to provide the second attachment to the current leads. These consist of NbTi conductors which are soldered to the joint. The ends of these leads and the end of the conductor are tinned with solder so it is easier to mount the joint in the testing holder. A finished joint is shown in figure 3-4.

## 3.2 Joint Testing

### 3.2.1 Apparatus

The apparatus for testing the joints consists of three major parts:

1. Dewar, magnet, and magnet supply;
2. Joint holder and joint supply;
3. Instrumentation.

This apparatus allows joints to be tested in the presence of field which can range from 0 to 3 T and with currents up to 500 A. The voltage versus current plot for each joint is recorded on an X–Y plotter.

#### Dewar, Magnet, and Supply

This part of the apparatus provides the liquid helium temperature and magnetic field required for testing the joints. The dewar has an inside diameter of 150 mm. The magnet used to provide the field was built in this lab from NbTi conductor and can provide up to 3 T at the center of its 25 mm bore. The magnet produces 1 T per 18 A of current through it. Current is provided to the magnet by a supply which can provide a maximum current and voltage of 100 A and 60 V. It is important to use a very high quality power supply with as little noise as possible. Noise produces fluctuations in the magnetic field, which in turn induce voltage fluctuation in the

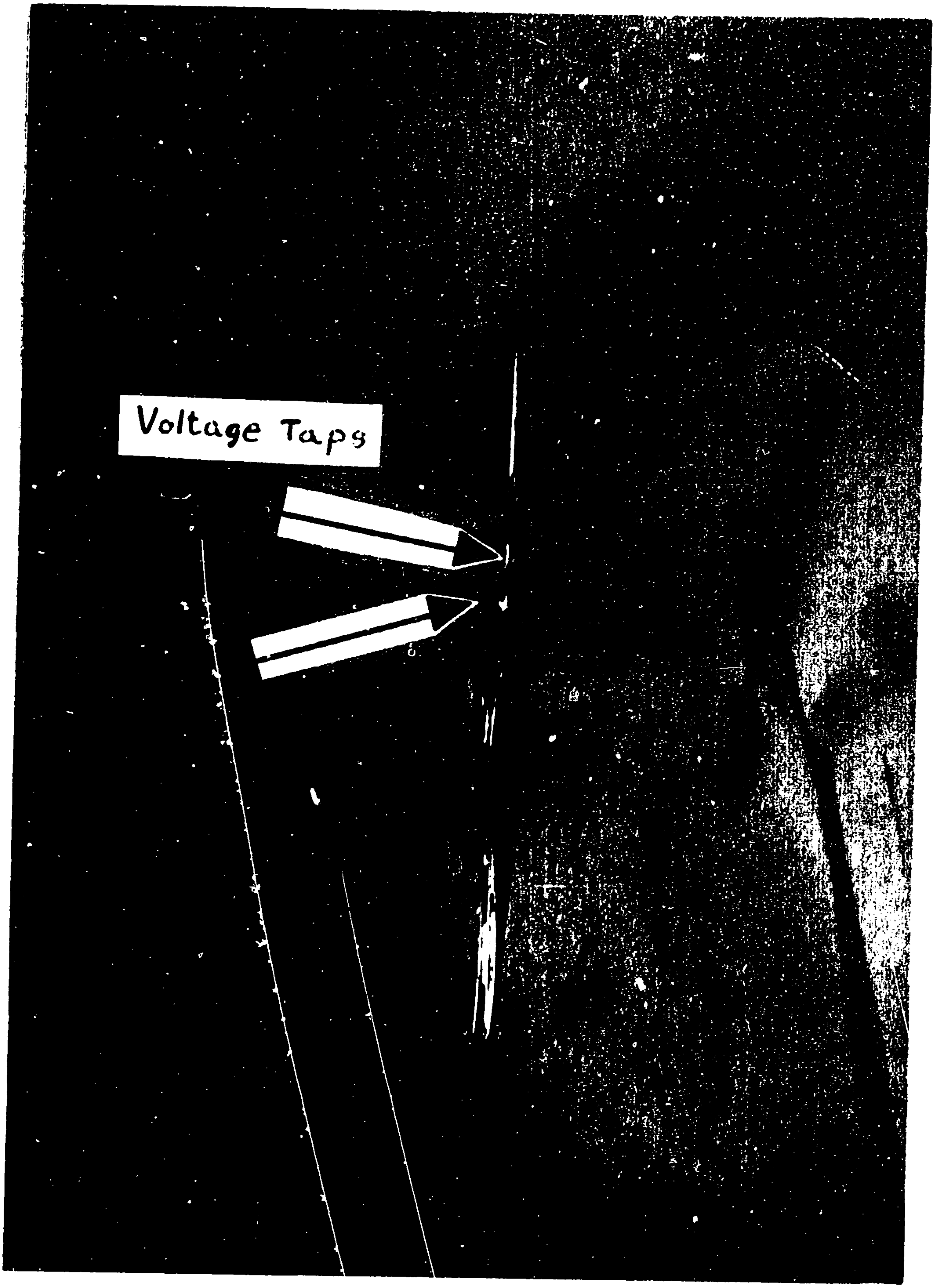

Figure 3-4: Example of a finished joint.

sample being tested which severely reduce the sensitivity of the measurements. A schematic showing the dewar, magnet, and supply are shown in figure 3-5.

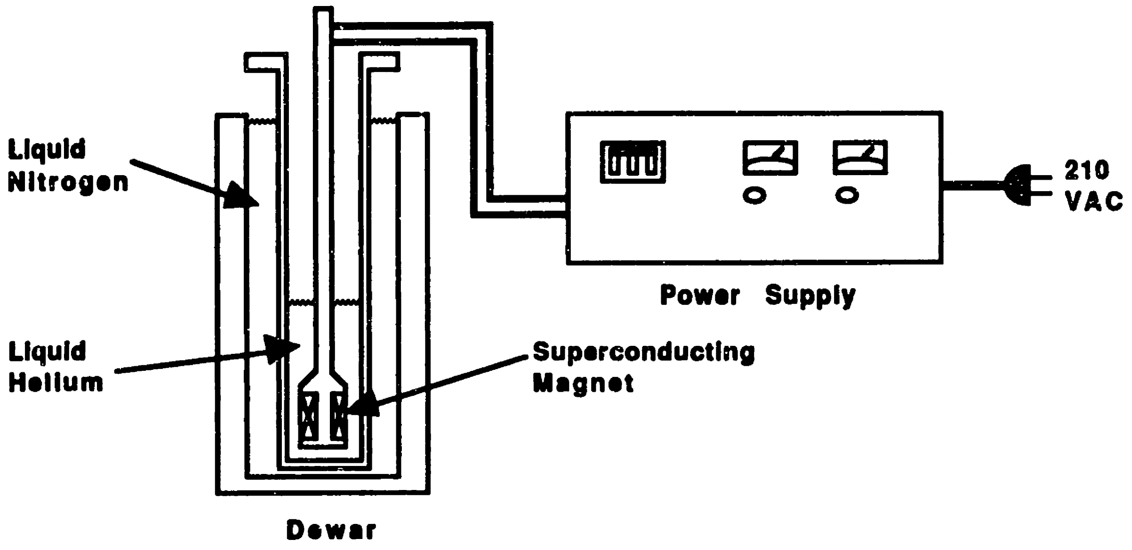

Figure 3-5: Schematic of dewar, magnet, and supply.

### Joint Holder and Supply

The joint holder serves two main purposes. It positions the joint inside the bore of the magnet and provides current leads for the joint. The holder is made from G10 and provides space for the joint to be attached to the two copper pads at the end of the current leads. The top copper pad can move freely so that damage to the joint from differential thermal contraction can be avoided. The current lead immediately above the top copper pad is very flexible fine copper cable to allow the top copper pad to move. The joints are held in place by attaching their leads to the copper pads with low temperature indium solder. The detail of the joint holder is shown in figure 3-8. A 500 A current source is used for testing the joints. A shunt resistor in series with the current leads is used to measure the current. A schematic of this part of the apparatus is shown in figure 3-7.

Figure 3-6: Joint holder detail.

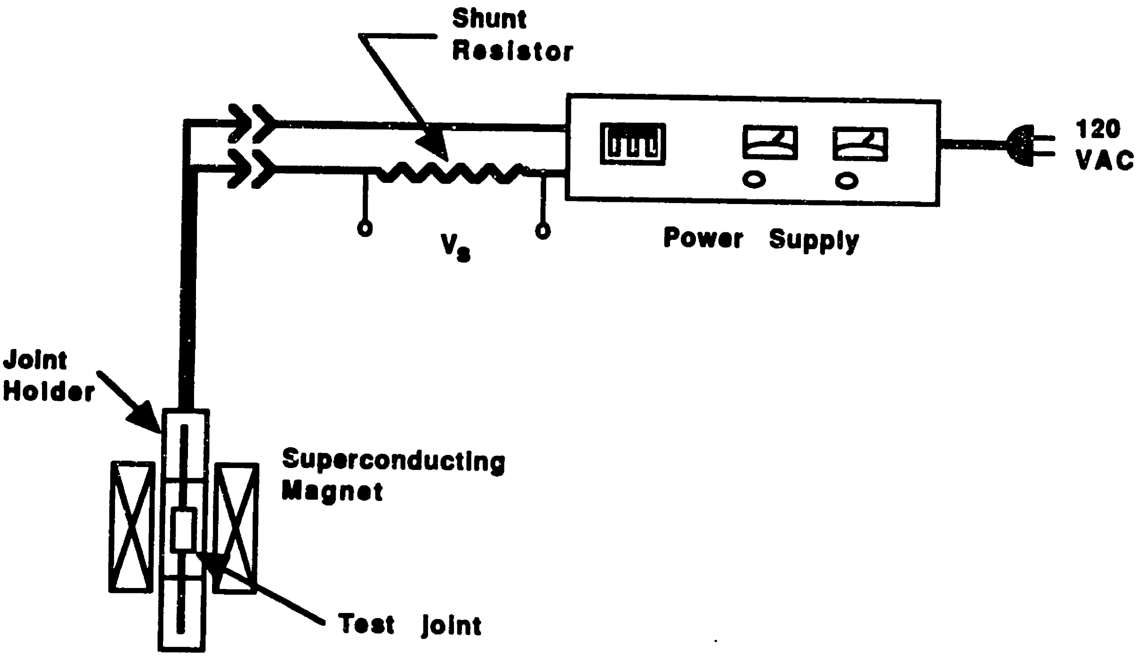

Figure 3-7: Schematic of joint holder and supply.

### Instrumentation

The joint is instrumented with voltage taps across the joint area. The potential across these taps is amplified, filtered and displayed as the vertical channel on an X–Y plotter. The horizontal channel records the current through the joint. The joints critical current is determined form the V-I plots.

The voltage taps are positioned as shown in figure 3-4. Since no current transfer resistance was observed, the placement of voltage taps was considered adequate. The thin copper wire used for the voltage taps is twisted together and brought out of the top of the apparatus for connection to the amplifier.

The amplifier used for voltage measurement has built-in filters to limit noise on its most sensitive setting (gain equals $10^6$). The output of the amplifier is connected to the X–Y plotter. In most instances the plotter was set to display 0.1 $\mu$V per cm vertically and 38 A per cm horizontally. The instrumentation setup is shown schematically in figure 3-8.

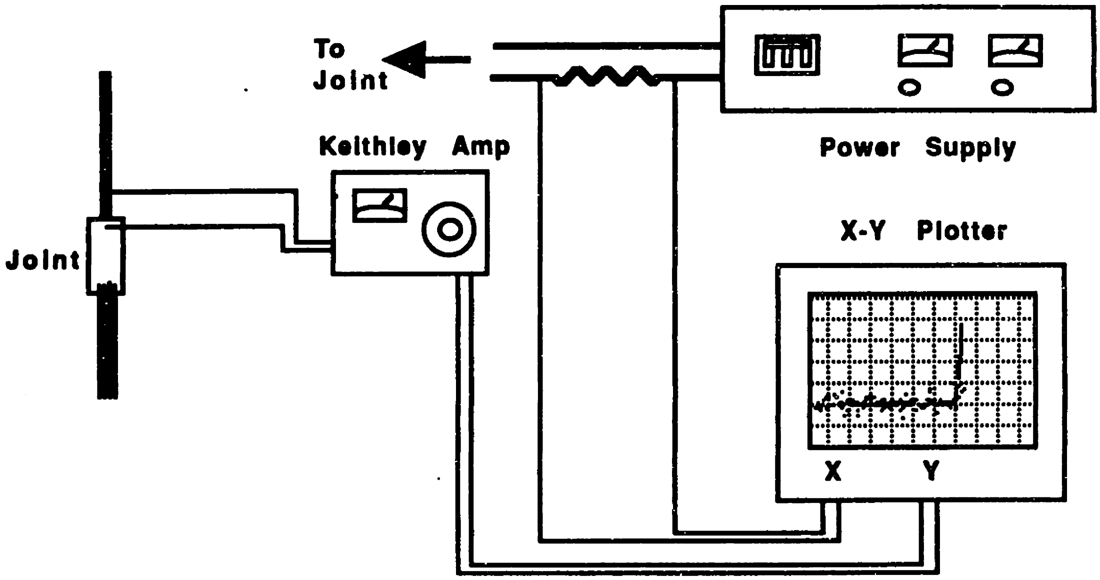

Figure 3-8: Instrumentation schematic.

## 3.2.2 Procedure

### Preparation

The first step in the testing procedure is to prepare the dewar and magnet assembly. The dewar is evacuated and the LN shield is charged. A small amount of LN is placed inside the dewar and then the opening is plugged with a cloth. The dewar is left in this condition for one or two hours while other parts of the apparatus are prepared. Once the dewar has been allowed to precool, the magnet is precooled in LN and then inserted into the dewar. Styrofoam plugs provided insulation around the opening of the dewar. After any remaining LN is removed, the transfer of helium can begin. The level of helium in the dewar is always monitored to make sure it never drops too low. The magnets supply can be connected and energized to see that the magnet is superconducting before beginning the testing.

### Testing

Once the dewar, magnet, and supply have been prepared, joints can be tested. With a joint mounted in the holder, the assembly is precooled in a bucket of LN. The holder and joint are then slowly lowered down into the bore of the magnet. Inserting the joint slowly allows the cold helium vapor to cool the joint below 77 K and reduce the amount of helium boiled off during insertion. Once the joint is positioned in the center of the magnet bore, the current leads can be connected to the joint supply and the voltage taps can be attached to the amplifier.

To test the joint, its current is slowly increased. This produces the V–I plot on the plotter. The current must be increased slowly because the amplifier filters have a very slow response. The joints can experience a very rapid transition to the normal state with little or no warning. Stopping the current quickly after a normal transition can reduce the amount of helium boiled off.

The test for each joint can repeated at any field between 0 and 3 T by changing the current in the magnet. After one joint has been tested, the joint holder can be removed, a new joint mounted, the assembly precooled, and the new joint tested. Each joint can be tested in a matter of minutes and requires only a little helium for cooldown.

This procedure allows the testing of several joints in succession in a relatively short amount of time. Joints of several different configurations could be tested to compare their performance. In addition, the same joint could be measured repeatedly after several thermal cycles between room temperature and 4.2 K to test for any degradation in performance. The descriptions of the actual joints tested and the results and conclusions from those tests are included in Chapter 4.

# Chapter 4

# Experiment and Results

This chapter discusses the different alternatives for building joints which were explored and how they led to the successful method. After each joint was built, it was tested against its performance criterion of 300 A in 3 T field. Some joints were also subjected to repeated thermal cycling and tested repeatedly for any degradation in performance. Complete results are summarized in this chapter. Conclusions and suggestions for future work are also presented.

## 4.1 Alternative Methods

To formulate alternatives to be investigated for joint making, all methods discussed in Chapter 2 were considered. It was decided to start with the method used by Williams for the 14.2 T NMR magnet [5]. Since Williams developed it at FBNML, the decision was reasonable. Several other unsuccessful variations of Williams method were also tried before arriving at the successful method described in Chapter 3.

### Adaptation of the Williams Method

The Williams method was successfully used in a 14.2 T NMR magnet. The main difference between the 14.2 T magnet and the 17.6 T magnet, currently being built, is the shape of the conductor. In the 17.6 T magnet the conductor has rectangular cross section, while the conductor in the 14.2 T magnet has round cross section.

The initial joints were thus made by broaching a rectangular hole in the composite piece for conductor. The conductor could be inserted with a specially modified cold welding device. The cold welder, normally used to make a butt weld between two round metal wires, was modified to allow it to grip a rectangular conductor and force it into the composite piece. This method was fraught with difficulties and never produced satisfactory results.

The main problems were in trying to adapt the previous method to the rectangular conductor. The modified cold welder dies could not grip the conductor with enough force to prevent it from slipping. Therefore, the amount of force which could be used to make the joint was much less than in the previous case. A second problem lie in the broaching operation. It was very difficult to make an adequate hole in the composite without breaking the broaching punch. Predrilling the hole to reduce the amount of material to be removed by the broaching punch did not help the problem much. The third problem was that the conductor was not gripped very tightly in the hole after heat treatment. The conductor must be supported in the hole to minimize force transmission to the joint. The joint is very weak and will break if it experiences much force at all. The conductor was made to fit as tightly as possible before heat treatment but afterwards there was not enough grip to hold the conductor in place while the joint was being prepared for testing.

### Split Composite

Further variations of the Williams method consisted of cutting the composite into two pieces and machining a groove in one half to accept the conductor. The conductor was inserted into the groove and moved up against the bottom to form a butt joint. The conductor for these joints was prepared by filing and polishing one end of the conductor. This was to provide a flat surface where the exposed ends of the filaments could contact the composite piece and form a butt joint.

Several of these joints were built but none were successful. The split composite made disassembly of the joint after testing easier. When these joints were disassembled, most showed a gap at the butt joint location. The gap forced current to flow through the conductor matrix to reach the composite, causing the joint to be resistive.

### Vertical Heat Treatment

In an attempt to maintain the butt contact in the split-composite method, a third variation was tried. In this method, the composite pieces were prepared exactly like the previous method. The conductor had a very small length of filaments exposed, typically about 1.0 mm. The joints were then held vertically in a fixture to maintain a force that kept the conductor end pressed against the composite during heat treatment.

A few good joints were produced by this method. However, the success was limited and not repeatable. Post-testing inspection revealed that the best joints had a greater amount of exposed filaments in contact with the composite. This method was abandoned because of the difficulty in applying it to the actual construction environment.

### Clamped-Filament Method

The clamped-filament method, described in Chapter 3, has proved the most successful. This method, at the present time, appears to be the most promising for use in the 17.6 T NMR magnet.

## 4.2 Experiments with Clamped Filament Joints

In all, 32 joints were produced for this project using the clamped-filament method. Several different configurations of the conductor and composite have been tried to see what optimize the method. The length of the conductor gripped by the the composite is one important variable. The length of the exposed filaments in the recessed area in the composite is another variable that determines joint performance. Refer to figure 3-4 for an example of the machined composite piece before joint assembly. The recessed area is 0.13 mm deep and 5.0 mm wide. The groove for the conductor provides an interference fit of 0.5 mm across the small dimension (clamping direction) and is

Table 4.1: Specifications of the $Nb_3Sn$ conductors in the 17.6 T NMR magnet

| Coil Number | Cross Section in (mm) |
|---|---|
| 1 | 3.43 × 1.37 |
| 2 | 2.79 × 1.50 |
| 3 | 2.77 × 1.24 |
| 4 | 2.74 × 0.99 |
| 5 | 2.03 × 1.19 |
| 6 | 1.98 × 0.94 |
| 7 | 1.75 × 1.02 |

0.5 mm wider than the conductors large dimension. The cross sectional dimensions of the seven $Nb_3Sn$ conductors in the 17.6 T NMR magnet are given in table 4.1. In order to simplify tabulation and comparison of the dimensions of the different joints, a standardized dimensioning system was used. The conductor and composite dimensions used to describe the joints are shown in figure 4-1. The dimension C is required to prevent the composite from shearing off the filaments during clamping. Some joints were built without this space before its importance was realized. To take advantage of the space care must be exercised to keep the conductor from sliding to far up the groove. The end of the matrix should not pass the beginning of the recessed area (*e.g.* the length of conductor in the composite should not exceed $A - C$). Some of the joints were built with extra tin powder added to the recessed area to help the exposed niobium filaments react during heat treatment.

## 4.3 Results of Testing

A two-step testing procedure was used for each joint. In the first step—performance test—the joint was tested according to the procedure in Chapter 3. The second step—reliability test—tested some joints several times after repeated thermal cycling. The purpose of the reliability test is to ensure that the joints are capable of withstanding several thermal cycles without failure. Since most thermal contraction occurs by 77 K,

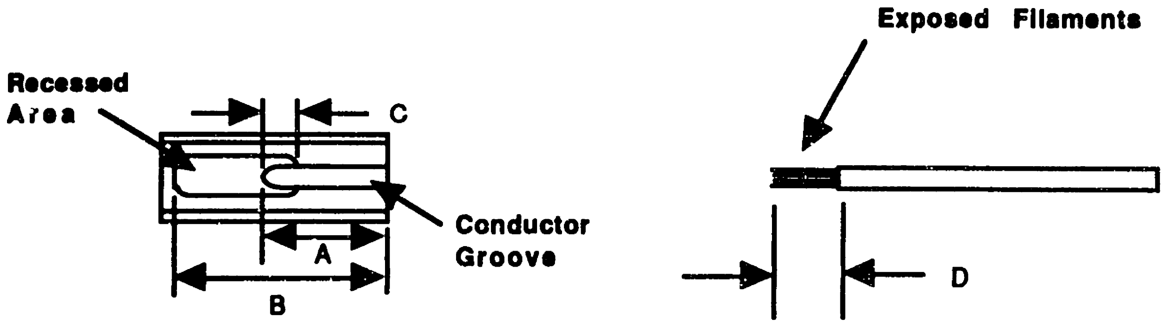

Figure 4-1: Standardized Dimensions for the Conductor and Composite. The different values of A, B, C, and D for each joint are given in table 4.2.

repeated cooldown in LN was considered sufficient for the reliability test. It is critical that the joints continue to perform well after several thermal cycles. The magnet will assuredly experience thermal cycling and new joints cannot be placed on the reacted magnet. The V–I plots, of selected joints, from performance and reliability tests are given in Appendix B. Tables 4.2 and 4.3 summarize the results of all the joints that were built and tested using the methods described in this thesis.

The results given in Appendix B show the V–I plots for joints which carried more than 200 A in zero field. These were the only joints tested in field. In all of the plots shown in Appendix B, the sensitivities are 0.1 $\mu$V/cm vertically and 38 $rmA$/cm horizontally. Since the plots have been reduced during reproduction the sensitivities are shown on each page. The plots in Appendix B include three joints, #4, #25, and #28, which carried more than 300 A in 3.0 T. Also included are the results for two joints tested for reliability. Each was cycled 20 times and showed no degradation of performance.

The critical current for each joint was found in the following way. First, fit a line to the data in the region before the superconducting-to-normal (SN) transition. Then draw a second line parallel to the first and 50 nV above it. A threshhold voltage of 50 nV is chosen because it is roughly 1 to 2 times the noise level in each plot. The point where the V-I plot intersects the second line defines the critical current.

To determine if the joints meet the 7 nV performance criterion, the $n$-index must be considered. The $n$-index is a property that determines the superconductor's V–I characteristics. It is defined by:

$$\frac{V}{V_0} = \left(\frac{I}{I_0}\right)^n \qquad (4.1)$$

The voltage $V_0$ is chosen to be some arbitrary small value and $I_0$ is the current corresponding to $V_0$. By selecting several pairs of $V$ and $I$ from the plot and performing a least squares analysis, the value of $n$ can be determined. Large values of $n$ indicate a sudden transition to the normal state whereas small values of $n$ indicate gradual transitions. The $n$-index is also a function of magnetic field. Typical values of $n$ for multifilamentary $Nb_3Sn$ conductors are 30–40. In joints such as #4 and #25 where the critical currents greater than 500 A in 3.0 T, an $n$-index greater than about 3.5 is all that is required to ensure that the voltage at 300 A in 3.0 T is less than the required 7 nV. All of the joints tested showed an $n$-index of greater than 3.5. However, joint #28, whose critical current is only 315 A, an $n$-index of more than 40 would be required to ensure that voltage in the joint is less than 7 nV at 300 A. Only a few joints had $n$ values that were greater than 40. Clearly, joints #4 and #25 are more desirable than #28 since the margin of safety is higher.

In some joints, a small resistance was measured prior to the SN transition. It is not clear if the resistance is an actual property of the joint or a result of the measurement technique. There are several possible causes for the resistance. The resistance could be due to current transfer in the conductor between the joint holder and the joint. Interfacial resistance between the two halves of the composite could also cause a voltage to appear across the voltage taps. More experiments are necessary to determine the cause of the pre-transition resistance.

In the reliability tests, joints #28 and #32 were selected because they had the highest critical currents under 500 A. These joints allowed detection of any change in the critical current after thermal cycling. If, for example, joint #4 had been used for the reliability test, then the critical current could only have been measured if it dropped below 500 A. It would have been possible for degradation in the performance to go undetected.

Table 4.2: Summary of Performance Test Results.

| Joint | Dimensions (mm) | | | | Critical Current (A) | | | Conductor | Extra |
|---|---|---|---|---|---|---|---|---|---|
| Number | A | B | C | D | 0.0 T | 1.5 T | 3.0 T | Number | Tin |
| 1 | 7.6 | 20.3 | 1.6 | 12.7 | 45.6 | – | – | 7 | yes |
| 2 | 7.6 | 20.3 | 1.6 | 12.7 | >500 | 346 | 236 | 6 | yes |
| 3 | 7.6 | 20.3 | 1.6 | 12.7 | 289 | 194 | 137 | 7 | no |
| 4 | 7.6 | 20.3 | 1.6 | 12.7 | >500 | >500 | >500 | 6 | no |
| 5 | 12.7 | 0.0 | 0.0 | 12.7 | 0.0 | – | – | 7 | no |
| 6 | 12.7 | 0.0 | 0.0 | 12.7 | 76.0 | – | – | 6 | no |
| 7 | 12.7 | 0.0 | 0.0 | 12.7 | 119 | – | – | 7 | yes |
| 8 | 12.7 | 0.0 | 0.0 | 12.7 | – | – | – | 6 | yes |
| 9 | 7.6 | 24.1 | 0.0 | 16.5 | 91.2 | – | – | 6 | no |
| 10 | 7.6 | 24.1 | 0.0 | 16.5 | 270 | 144 | 98.8 | 7 | no |
| 11 | 7.6 | 24.1 | 0.0 | 16.5 | – | – | – | 6 | yes |
| 12 | 7.6 | 24.1 | 0.0 | 16.5 | 0.0 | – | – | 7 | yes |
| 13 | 7.6 | 24.1 | 0.0 | 16.5 | 0.0 | – | – | 6 | no |
| 14 | 7.6 | 16.5 | 0.0 | 8.9 | 410 | 262 | 163 | 7 | no |
| 15 | 7.6 | 16.5 | 0.0 | 8.9 | 91.2 | – | – | 6 | yes |
| 16 | 7.6 | 16.5 | 0.0 | 8.9 | 0.0 | – | – | 6 | yes |
| 17 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 18 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 19 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 20 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 21 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 22 | 7.6 | 20.3 | 2.5 | 12.7 | 46.0 | – | – | 6 | no |
| 23 | 7.6 | 20.3 | 2.5 | 12.7 | 19.0 | – | – | 6 | no |
| 24 | 7.6 | 20.3 | 2.5 | 12.7 | 334 | 190 | 144 | 6 | no |
| 25 | 7.6 | 20.3 | 2.5 | 12.7 | >500 | >500 | >500 | 6 | no |
| 26 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 27 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 28 | 7.6 | 20.3 | 2.5 | 12.7 | >500 | 471 | 315 | 6 | no |
| 29 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 30 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 31 | 7.6 | 20.3 | 2.5 | 12.7 | 0.0 | – | – | 6 | no |
| 32 | 7.6 | 20.3 | 2.5 | 12.7 | >500 | 361 | 255 | 6 | no |

Table 4.3: Summary of Reliability Test Results.

| Joint Number | Critical current at 3 T after $n$ cycles (A) | | | | |
|---|---|---|---|---|---|
| | $n = 0$ | $n = 5$ | $n = 10$ | $n = 15$ | $n = 20$ |
| 28 | 315 | 315 | 323 | 312 | 289 |
| 32 | 255 | 251 | 262 | 247 | 255 |

## 4.4 Conclusions

Several conclusions can be drawn that are important to the future development of this joint making method. The tests in this thesis were essentially a proof of the concept of the clamped filament method. Suggestions for future work are given in the next section.

The first and most important conclusion is that this method can produce joints which meet the performance criterion (300 A in 3 T with less than 7 nV) and are reliable. Joints with performance like that of #4 and #25 are desirable since they have a large margin of safety with respect to the 7 nV requirement. The reliability tests on joints #28 and #32 show that the joints can maintain their performance after repeated thermal cycling. Several other joints were tested second and third times and now decrease in critical current was ever observed. Joints #4 and #25, in a separate test, had the two halve of their composite pieces split apart and the piece retaining the conductor was tested as if it were a normal joint. Even after this abuse, the joints showed no decrease in performance. It is almost certain that with further work this method could be used in the construction of the 750 MHz NMR magnet.

Experience in building the joints in this thesis indicates that there are some important factors to be considered when refining the technique of this method. It is clear from the data that no additional tin was required for successful joints. Since tin has such a low melting point it is possible that the tin flowed out of the joint during heat treatment. Evidence of escaped tin was always seen in the retort after heat treatment. It is also important not to damage the filaments during assembly of the joints. Joints #5 through #16 were disassembled after testing and broken filaments were discovered. The damage was apparently due to the composite shearing off the filaments during assembly because adequate clearance was not provided. Joints #17 through #24 were exposed to air during heat treatment and their performance was generally poor. These joints appeared to be oxidized on the surface and inside. Experience has shown that even small amounts of air in the retort can have adverse effects on the heat treatment procedure.

There are some conclusions which are important to the testing of future joints. It is possible that the two sides of the joint do not perform equally. This is shown by the testing of both sides of joint #28. This is a very limited conclusion, but it should be investigated in future tests because it could have some bearing on the construction of joints in the future. Also, since the SN transition of the most desirable joints is beyond the range of the apparatus in this experiment, it is concluded that equipment for testing at higher fields and currents would be helpful in the future.

An interesting observation regarding all the joints except #4 and #25 is that the critical current decrease rapidly with increasing field. It is possible that some superconducting material besides $Nb_3Sn$ is formed in the joint area. This material, if it had a low critical field, might effect the joints performance in a way consistent with these results. This is only conjecture and should be examined carefully in future tests.

## 4.5 Suggestions for Future Work

While the clamped-filament method for building superconducting joints has been shown to be valid, some work is still required to demonstrate that the method is *totally* suitable for the 17.6 T magnet. The first task is to refine the technique by continuing to build and test joints. Specifically, special attention should be given to ensuring that the composite and conductor filaments are free of any contaminates or oxide layers. To help determine what differentiates good and bad joints some characterization of the joint is required. For example, sectioning the joints and making photomicrographs can show if $Nb_3Sn$ is forming and if it is being damaged. Tests to determine what substances exist in the joint would be helpful as well. The analysis of the joints will help refine the fabrication technique and as more joints are built and tested, confidence in the method will become more certain and the highest reliability possible can be achieved.

Future testing of the joints should be continued as described in this thesis with the following additions. Once the performance level of joints #4 and #25 can be routinely achieved, then the joints should be tested in higher fields and with higher currents to determine the limits of there performance. The final test of the joints should be to build actual size prototypes with the NbTi-to-composite joint included (refer to section 2.2.3). Theses joints should be tested in persistent mode using the technique of Iwasa and Leupold [15]. This method measures the decay of the persistent current and resistances as low as $10^{-14}$ $\Omega$ can be detected. The goal of all future work should be to verify that actual joints for the 17.6 T NMR magnet can be built using the clamped filament method.

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# Appendix A

This appendix contains the following US patents as examples of joint making methods.

**3,422,529** Describes a method of joining NbZr monofilament conductors to each other.

**3,527,876** Describes a method of joining NbZr monofilament conductors to a NbTi plate.

**4,713,878** Describes a method of joining NbTi multifilamentary conductors to each other using superconducting solder.

**4,901,429** Describes a method of joining NbTi multifilamentary conductors to each other using ultrasonically vibrated molten metals to etch and join them.

US Patent 3,422,529

# United States Patent Office

3,422,529
Patented Jan. 21, 1969

3,422,529
## METHOD OF MAKING A SUPERCONDUCTIVE JOINT

James M. Neding, Canoga Park, Calif., assignor to North American Rockwell Corporation, a corporation of Delaware

Filed Dec. 9, 1963, Ser. No. 329,311
U.S. Cl. 29—599 6 Claims
Int. Cl. H01s *4/00;* H01r *43/04*

My invention relates to a method of making a junction between two pieces of superconducting wire, and more particularly to a method of making such a joint which will be superconductive.

Superconductivity is the property of certain materials at cryogenic temperatures approaching absolute zero to carry extremely large currents in strong magnetic fields without power dissipation. Such materials, at temperatures below a certain critical temperature, $T_c$, have no electrical resistivity, and therefore no $I^2R$ losses. This phenomenon has been experimentally verified. Coils of such materials in liquid helium baths, with currents induced by such means as withdrawing a permanent magnet from within the coil, have carried the resulting currents for periods of two years without any voltage drop. The factors affecting superconductivity of such materials are the interrelation of magnetic field strength H, critical current density $J_c$, and critical temperature $T_c$. The magnetic field strength, applied externally or generated by a current in the superconductor, limits superconductivity to below certain temperatures and current densities. Similarly, at a given field strength, an increase in temperature and/or current density can terminate superconductivity. The large current-carrying capacity of superconductors provides the basis for very compact, super-powerful magnets which can be used in numerous applications where strong magnetic fields are required, for example, in lasers, masers, accelerators, and bubble chambers.

Superconducting devices display the tendency, for reasons not thoroughly understood but believed to include application of excessive current or by local heating, to undergo a transition from the superconductive state to a normal conductive state, after which a superconductive condition can be reestablished. This transition, which is called an SN transition, causes large induced voltage drops to appear across the superconducting magnet when the strong fields collapse. Such voltage bursts may damage superconducting solenoids in addition to rendering them inoperative for short periods.

The tendency to undergo SN transitions is particularly pronounced at junctions between superconducting wires. Such junctions are frequently necessary in the fabrication of relatively large solenoids because of limitations on the length of superconducting wire which can be drawn in one section. It has been necessary to use wire ranging in lengths of from about 1,000 feet to 7,000 feet, due to difficulties in manufacturing longer lengths of the relatively brittle wire. Further, the cost of wire increases proportionately to an increase in length. Solenoids which require greater lengths of wire than heretofore obtainable have been constructed making joints between shorter lengths of wire. Such joints have been made by bringing the ends of wires to be joined outside of the solenoid but still in the coolant bath. Pressure-type connections are made with clamping screws on terminal strips or by soldering with such metals as copper-brass. This method of making connections is complicated and difficult, and such junctions are frequently non-superconducting or display a greater tendency to undergo SN transitions. The method is particularly disadvantageous for the fabrication of very large solenoids where several hundred thousand feet of wire are needed

and many junctions would have to be made in a low flux region. Unless the entire solenoid is superconducting, a persistent flow of current will not be maintained after the power source is turned off, and hence the practical value of the solenoid is reduced.

An object of the present invention, accordingly, is to provide an improved method of joining sections of superconducting wire.

Another object is to provide a method of joining sections of superconducting wire, wherein the resulting joint is superconductive.

Another object is to provide a method of joining superconducting wire, wherein the junction will pass as much current as a solid piece of the same wire in a given magnetic flux field.

Still another object is to provide a junction between two pieces of superconducting wire which can be wound on a solenoid without displaying any greater tendency to undergo SN transitions than the parent metal.

The above and other objects and advantages of the present invention will become apparent from the following detailed description and the appended claims.

In the drawings, FIG. 1 is a schematic view of the joint components prior to assembly, and FIG. 2 is a schematic view of a completed joint.

In accordance with the present invention I have provided a method of making a superconductive joint between two sections of superconducting wire, which comprises twisting together ends of the wire, placing the resulting twisted section in a metal sleeve, and then cold pressing the resulting assembly.

The essential aspects of the present invention are illustrated in the drawing. In FIG. 1, two separate lengths of superconducting wire 2 and 4 are twisted together to form twisted section 6. The twisted end 6 is inserted in a metal sleeve 8 and the resulting assembly pressed at sufficient pressure to yield the final superconductive joint 8*a*. In addition to the embodiment shown where the superconducting wires are inserted into the cylinder from the same end, they may also be inserted from opposite ends.

The sleeve is pressed under sufficient pressure to bring about maximum contact between the twisted ends of the wire, which insures that the ends will not separate during thermal cycling, handling operations, or the like. The cold pressing also allows the metal of the cylinder to cold flow around the twists of wire until maximum contact is made between the twist, and between the twist and the cylinder. Joints made in this manner are superconductive, will pass large currents in kilogauss magnetic fields, may be wound directly onto solenoids, and will pass as much current as a single length of the same wire without sustaining SN transitions.

Experiments have shown that joints prepared under identical conditions, but without the twists, with the wires lying parallel to each other, are either non-superconducting or undergo SN transitions at relatively low current values. It is believed that this results from slight separations of the wires, which permits flow of sleeve metal therebetween, or from other conditions resulting in non-superconducting transition sections.

The two ends of the superconducting wire to be joined are first twisted together. The ends are generally even to each other, and the number of turns may vary while achieving a satisfactory joint. It is found, however, that the joint should have at least about three turns, placed about $\frac{1}{16}$ inch apart. For example, when 10-mil wires are joined in a cylinder $\frac{3}{16}$ inch long, the twisted length inserted into the cylinder has at least three and preferably about four turns. The number of turns per unit length of wire will depend upon the thickness and physical properties of the alloy wires; too many turns of a relatively thin, brittle wire may cause it to break. For example, six or more turns of 10-mil Nb-25 Zr alloy wire over a $\frac{3}{16}$ inch length may cause fracture.

The twisted wire pair is then cleaned and inserted into the sleeve. The cleaning solution removes any oxide or organic film on the surface of the wire which might prevent complete contacting and formation of a superconductive joint. The common cleaning or pickling solutions known to the art may be used, such as a solution containing about 48 percent nitric acid, 2 percent hydrofluoric acid, and 50 percent water.

The cylinder or sleeve into which the twisted wire is inserted may be of either the same alloy as the superconducting wire or of stainless steel. The wall diameter of the cylinder should be such that it has adequate strength to maintain the wires under the compressive load imparted during cold pressing without fracturing. The axial hole through the cylinder need be of sufficient diameter only to permit ready insertion of the twisted wire. For example, for joining 10-mil wire, the cylinder may satisfactorily be $\frac{3}{16}$" long by $\frac{1}{16}$" O.D. with an axial hole 0.025" in diameter. The jacket metal may satisfactorily be of the same metal as the superconducting wire. However, since such metals are relatively brittle, any other nonmagnetic metal, such as stainless steel, having a tensile strength at least equal to that of the wire may be used as the sleeve material. Metals having higher ductility and lower tensile strength than the superconducting wire will cold flow axially and cause fracture of the wire, and may therefore not be used. The cylinder is cleaned in the same manner as the twisted wire prior to insertion of the pair therein.

The small cylinder is then slipped over the twisted pair so that two or three twists are inside the cylinder, and the assembly squeezed in a hydraulic press at a pressure sufficient to bring about maximum contact between the twists and between the twists and the cylinder. While the pressure applied to the assembly may satisfactorily vary, a pressure approaching the tensile strength of the superconducting wire is found to produce superior results and is, therefore, preferred. For example, in joining Nb-25 weight percent Zr alloy, which has a tensile strength of about 200,000 p.s.i., cold pressing at a pressure of about 170,000–200,000 p.s.i. is optimum. The pressure is maintained for a period sufficient to permit the cold flow previously mentioned. This is in the order of several minutes, and a period of about fifteen minutes is generally allowed for the pressing of the Nb-25 weight percent Zr wire at the before-indicated pressure. It is also found, however, that pressure on the joint in excess of the tensile strength of the wire has a deleterious effect and produces an inferior joint due to fracture of the wire. Accordingly, the pressing is preferably conducted at a pressure approaching but not exceeding the tensile strength of the wire.

The following example is offered to illustrate my invention in greater detail.

A junction between two pieces of 10-mil Nb 25% Zr superconducting wire was made by joining the two ends of the superconducting wire with the ends even each other, and twisting them together with four twists over a length of $\frac{3}{16}$ inch. The twisted wire was cleaned in a solution of 48% nitric acid, 2% hydrofluoric acid, and 50% water. A small rod of the same alloy was machined into a cylinder $\frac{3}{16}$" long by $\frac{1}{16}$" O.D. with an axial hole 0.025" in diameter, and was cleaned with the same cleaning solution. The sleeve was slipped over the twisted pair so that about three twists were inside the sleeve. The assembly was squeezed in a hydraulic press to a pressure of about 200,000 p.s.i. and allowed to remain under such pressure for about 15 minutes.

The resulting joint was tested in a 30,000 gauss magnetic field at liquid helium temperature; it passed more than 100 amperes without sustaining an SN transition. For comparison purposes, identical wires lying parallel to each other were pressed in the same cylinders under the same conditions. In tests under the foregoing conditions, SN transitions resulted at currents ranging from 5 amps to 25 amps.

The foregoing example is offered for purposes of illustration rather than restriction. Variations may be made by those skilled in the art without departing from the spirit of the present invention.

I claim:

1. A method of forming a superconductive joint between pieces of superconductive wire, which comprises twisting the ends of said wire together, inserting the resulting twisted pair into a small metal sleeve, and cold pressing the resulting assembly at a pressure approximately equal to the tensile strength of both the sleeve and superconductive wires until firm contact is made between the twists of wire and the sleeve.

2. The method of claim 1, wherein the metal sleeve is nonmagnetic.

3. The method of claim 1, wherein the sleeve is made from a metal selected from the class consisting of stainless steel and the same metal as the superconductive wire.

4. A method of joining two pieces of niobium-zirconium alloy superconductive wire, which comprises twisting the ends of the wire together, positioning a small nonmagnetic metal cylinder having a tensile strength at least equal to said alloy over the twisted pair so that a plurality of twists remain inside the sleeve, cold pressing the resulting assembly at a pressure of about 170,000–200,000 p.s.i. until firm contact is made between the twists and the sleeve.

5. The method of claim 4 wherein said alloy consists essentially of about 25 weight percent zirconium and the remainder niobium, and a pressure of about 200,000 p.s.i. is applied for a period of about 15 minutes.

6. A method of making a superconductive junction between two lengths of fine niobium-zirconium wire, which comprises twisting the two ends of the wire together with at least three turns spaced about $\frac{1}{16}$ inch apart, cleaning the resulting twisted pair, providing a small stainless steel cylinder with an axial hole sized to receive the twisted pair, cleaning said cylinder, inserting the wire into the cleaned cylinder so that the twists are positioned inside the cylinder, cold pressing the resulting assembly at a pressure of about 170,000–200,000 p.s.i. until complete contact is made between the turns of the twist and between the twists and the cylinder.

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U.S. Cl. X.R.

29—518, 628; 174—87

US Patent 3,422,529

Jan. 21, 1969 J. M. NUDING 3,422,529

METHOD OF MAKING A SUPERCONDUCTIVE JOINT

Filed Dec. 9, 1963

FIG. 1

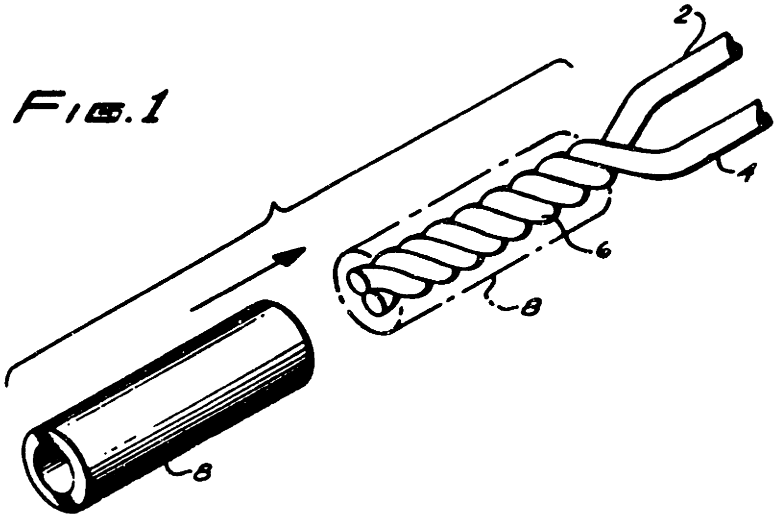
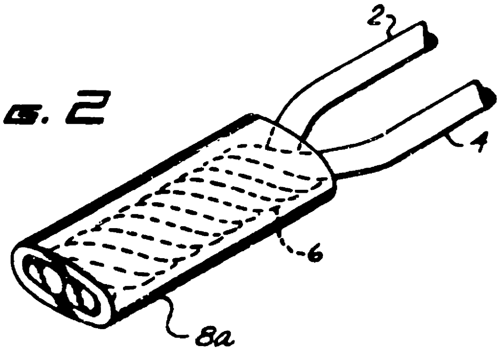


FIG. 2



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United States Patent Office

3,527,876

Patented Sept. 8, 1970

3,527,876

# ELECTRICAL CONNECTION BETWEEN SUPERCONDUCTORS

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Claims priority, application Switzerland, Oct. 23, 1969, 14,574

Int. Cl. H02g 15/08

U.S. Cl. 174—94 7 Claims

## ABSTRACT OF THE DISCLOSURE

An electrical connection between the end portions of two conductors made of hard-super-conductive material is formed by spot welding the conductor end portions to a plate also made from a hard-superconductive material in which the critical current-density has been increased by annealing. The hard-superconductive conductors are coated with copper which is removed at the places where the spot welds are made, and these copper coatings are electrically connected to a sheet of copper which forms an underlay for the hard-superconductive plate.

The invention relates to an electrical connection between the ends of two conductors whereof at least one is made of a hard-superconductive material.

The discovery of superconductive materials which do not lose their superconductivity even in the presence of fairly high magnetic field-strengths has made it possible in recent years to construct for the first time certain devices such for example as superconductive coils for generating high magnetic field-strengths. The best known of the superconductive materials of this kind, which are generally called "hard superconductors," takes the form of a niobium-zirconium alloy which, when made into wires or strips, is suitable for constructing coils with which values of magnetic induction of over 30 kg. may be generated. In the construction of such coils, a particularly difficult problem is involved in making electrical connections between superconductive conductor elements and between such elements and the current-source. In these devices, the connections in fact nearly always constitute points of minimum "critical current-density," and thus define the onset of transition to the normal conductive state.

In a known process for making a connection between two hard-superconductive wires which allow for sufficiently high current-density in the superconductive state in the presence of a magnetic induction of more than 30 kg., the wires to be connected are freed of their insulating or normal conductive surface layers, as distinguished from superconductor, and connected to one another with the aid of a clamping element whereof the clamping jaws are coated with a layer of a hard-superconductive material. Although such a clamped connection exhibits a sufficiently low electrical resistance of 10[illegible] Ω, which moreover has little dependence on the magnetic induction, the fact that it occupies a relatively large amount of space makes it unsuitable for many purposes such for example as connecting two conductors inside a coil.

According to another known process, the ends of the conductors are connected by spot-welding. Such connections have a very low electrical resistance of $\sim 10^{-9}$ Ω, but the welding produces structural changes in the ends of the conductors which considerably reduce the critical current-density at these points. Such connections can thus be used only at points of relatively low induction.

The invention is based on the problem of providing an electrical connection between hard-superconductive conductors which does not exhibit the said disadvantages of known connections.

According to the present invention there is provided an electrical connection between the ends of two conductors whereof at least one is made of a hard-superconductive material, the connection exhibiting an electrical resistance of less than $10^{-8}$ Ω in the presence of a magnetic induction of 30 kg., wherein the ends of the conductors are spot-welded to a plate made of a hard-superconductive material of the type in which the critical current-density has been increased by annealing.

The invention is explained, by way of example, with reference to the accompanying drawings wherein:

FIG. 1 is a plan view of the two conductors as connected together.

FIG. 2 is a transverse section on line II—II of FIG. 1, and

FIG. 3 is a graph showing the relation between the current in the connection and the magnetic transverse-field induction.

A connection between two hard-superconductive wires is illustrated in FIGS. 1 and 2. The wires 1, which are made of a niobium-zirconium alloy with 25% by weight of zirconium, are provided with a copper coating 2 which is removed at the ends of the wires where the connection is to be made. The ends of the wires are spot-welded to a plate 3. This plate 3 is made of a niobium-titanium alloy with 40% by weight of titanium, and is connected at the back to a copper sheet 4 extending beyond the plate 3, which sheet is soldered at the points 5 to the copper coatings 2 of the wires 1 for the purpose of forming a normal conductor shunt connection. FIG. 1 shows the outlines 6 of the spot-welds which each connect the ends of both wires to the plate 3.

The alloy used for the plate 3 is one of those hard-superconductive materials in which the critical current-density is increased during production by annealing after the last change in shape, and not by cold deformation as in the case of Nb-Zr alloys. Such alloys are preferably niobium-titanium alloys with 10–90% by weight of titanium. In order to improve their technological or physical properties, a tantalum additive of 0–30% by weight may be provided.

In order to prevent a transition from the superconductive state to the normal conductive state spreading from the plate 3, every cross-sectional area F of the plate 3 traversed by the current in one direction must be sufficiently large for the conduction $I \leq i_c \cdot F$ to be always fulfilled for the critical current-density $i_c$ of the plate material and for the maximum permissible current I.

In FIG. 3, curve A shows how the critical amount of current $I_c$ in the connection described depends on the magnetic transverse-field induction $B_\perp$. For comparison, curves B and C show the critical amounts of current for the uninterrupted wire and for a connection in which the ends of the wires are directly spot-welded to one another.

As will be seen, the critical amount of current $I_c$ is considerably greater in the case of the connection described (curve A) than in the case of the connection with direct welding of the ends of the wires (curve C).

We claim:

1. An electrical connection between the ends of two conductors whereof at least one is made of a hard-superconductive material, the connection exhibiting an electrical resistance of less than $10^{-8}$ Ω in the presence of a magnetic induction of 30 kGauss, wherein the ends of the conductors are spot-welded to a plate made of a hard-superconductive material of the type in which the critical current-density has been increased by annealing.

2. An electrical connection according to claim 1, where-

in the plate is connected to an underlay of normal conductor material.

3. An electrical connection according to claim 1, wherein the hard-superconductive conductors comprise coatings of a normal conductor material which are removed at least at the spot-welds.

4. An electrical connection according to claim 3, wherein the normal conductor coatings on said hard-superconductive conductors are electrically connected to one another by a normal conductor piece.

5. An electrical connection according to claim 1 wherein said hard-superconductive conductors include copper coatings thereon which are removed at the spot welds and which further includes a copper sheet forming an underlay for said plate, said copper sheet being electrically connected to said copper coatings.

6. An electrical connection according to claim 1, wherein the plate is made of a niobium-titanium alloy with 10–90% by weight of titanium and an additive of 0–30% by weight of tantalum.

7. An electrical connection according to claim 1, wherein every cross-sectional area F of the plate traversed by the current in one direction is sufficiently large for the condition $I \leq i_c \cdot F$ to be always fulfilled for the critical current-density $i_c$ of the plate material and for the maximum permissible current I.

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DARRELL L. CLAY, Primary Examiner

U.S. Cl. X.R.

29—599, 628; 335—216

# US Patent 3,527,876

**Sept. 8, 1970** E. KARVONEN ET AL **3,527,876**

ELECTRICAL CONNECTION BETWEEN SUPERCONDUCTORS

Filed Sept. 23, 1968 2 Sheets-Sheet 1

Fig. 2

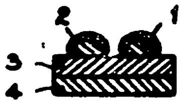
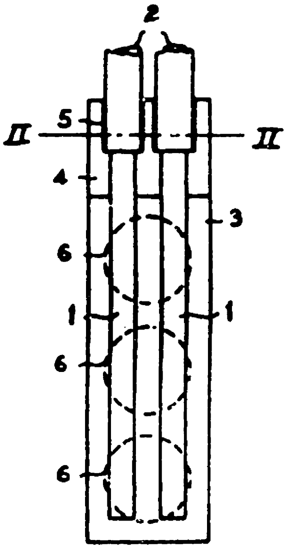


Fig. 1



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**Sept. 8, 1970** E. KARVONEN ET AL **3,527,876**

ELECTRICAL CONNECTION BETWEEN SUPERCONDUCTORS

Filed Sept. 23, 1968 2 Sheets-Sheet 2

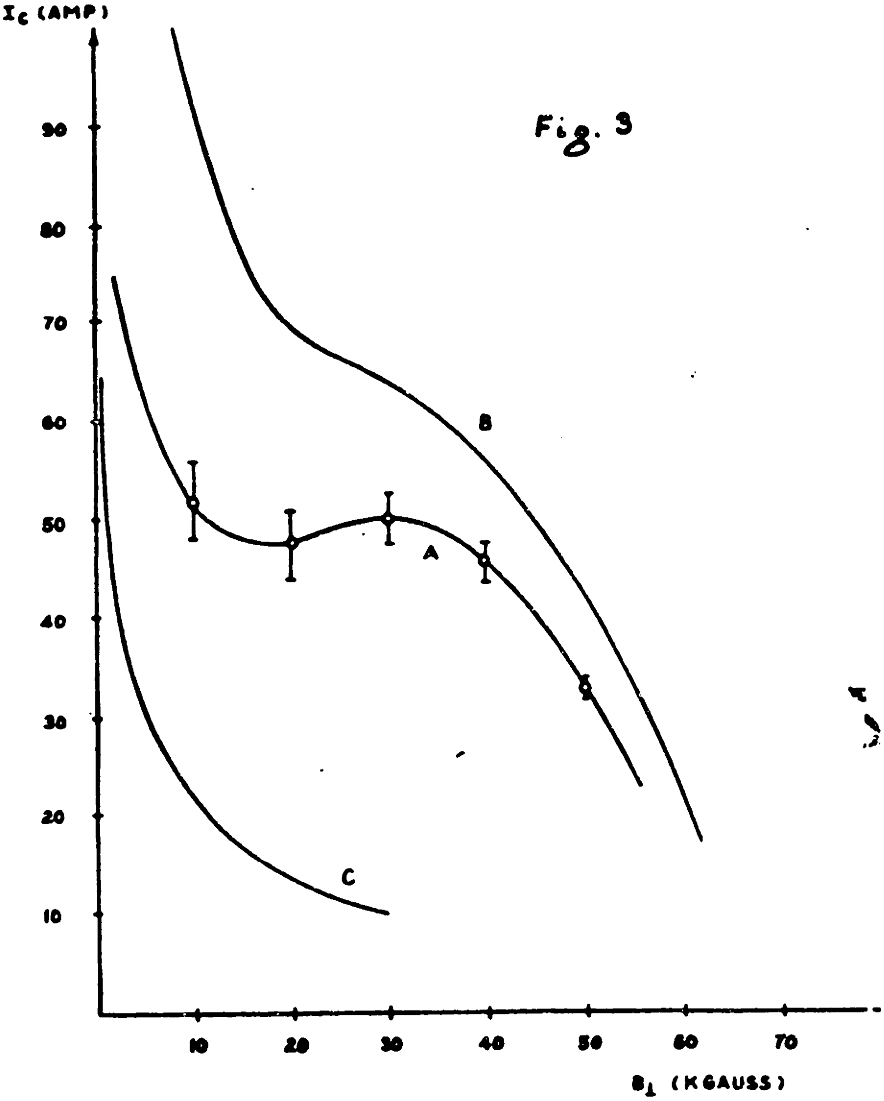

Fig. 3

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US Patent 4,713,878

**United States Patent** [19]

**Kumpitsch et al.**

[11] **Patent Number:** **4,713,878**

[45] **Date of Patent:** **Dec. 22, 1987**

[54] **MOLD METHOD FOR SUPERCONDUCTIVE JOINT FABRICATION**

[75] Inventors: **Robert C. Kumpitsch**, Johnstown; **James P. Retersdorf**, Gloversville, both of N.Y.

[73] Assignee: **General Electric Company**, Schenectady, N.Y.

[21] Appl. No.: **678,443**

[22] Filed: **Dec. 5, 1984**

[51] Int. Cl.$^4$ .................... **H01L 39/24**

[52] U.S. Cl. .................... **29/599**; 174/126 S; 174/128 S

[58] **Field of Search** .................... 29/599; 174/10, 70 R, 174/87, 90, 94 R, 94 S, 126 R, 126 S, 128 S

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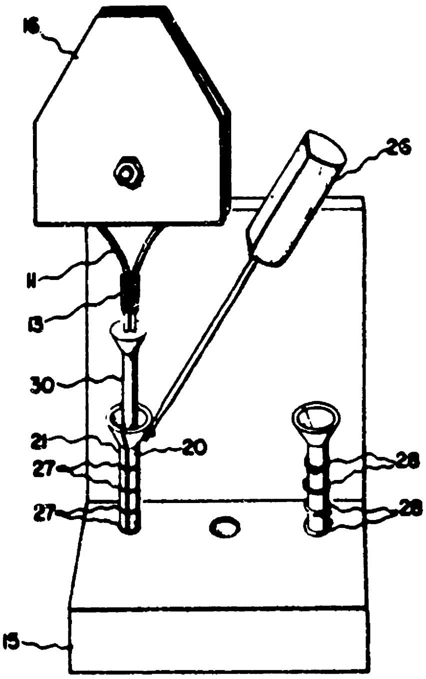
*Primary Examiner*—Howard N. Goldberg
*Assistant Examiner*—Carl J. Arbes
*Attorney, Agent, or Firm*—Lawrence D. Cutter; James C. Davis, Jr.; Marvin Snyder

[57] **ABSTRACT**

A method for joining multifilamentary superconductive wire comprises disposing the ends of the wires in a hot liquid metal stripping bath for removal of the metal matrix. In particular, in the present invention the ends of the wires to be joined are agitated within this bath to assure complete removal of the metal matrix. The liberated superconductive filaments are then disposed in a solder bath and then inserted into a mold which is filled with hot liquid superconductive solder which is allowed to solidify around the filamentary conductors after which the mold is removed.

**29 Claims, 4 Drawing Figures**



US Patent 4,713,878

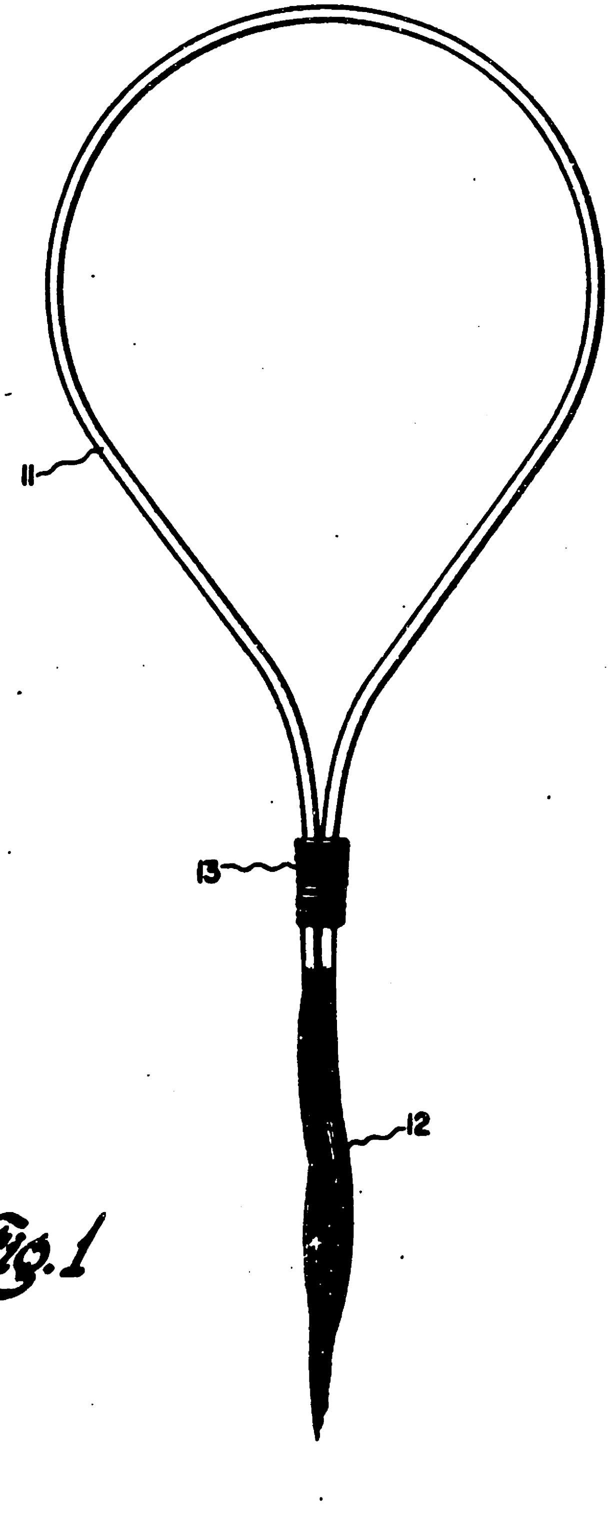


Fig. 1

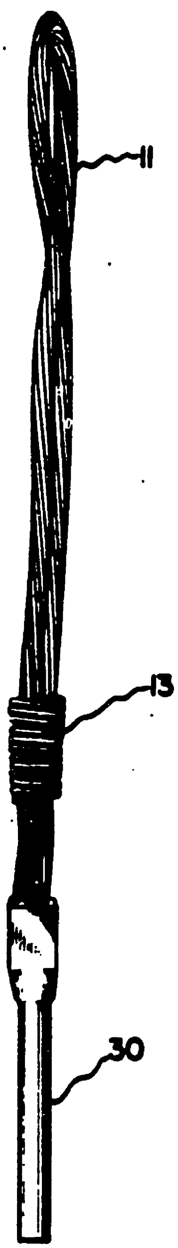


Fig. 3

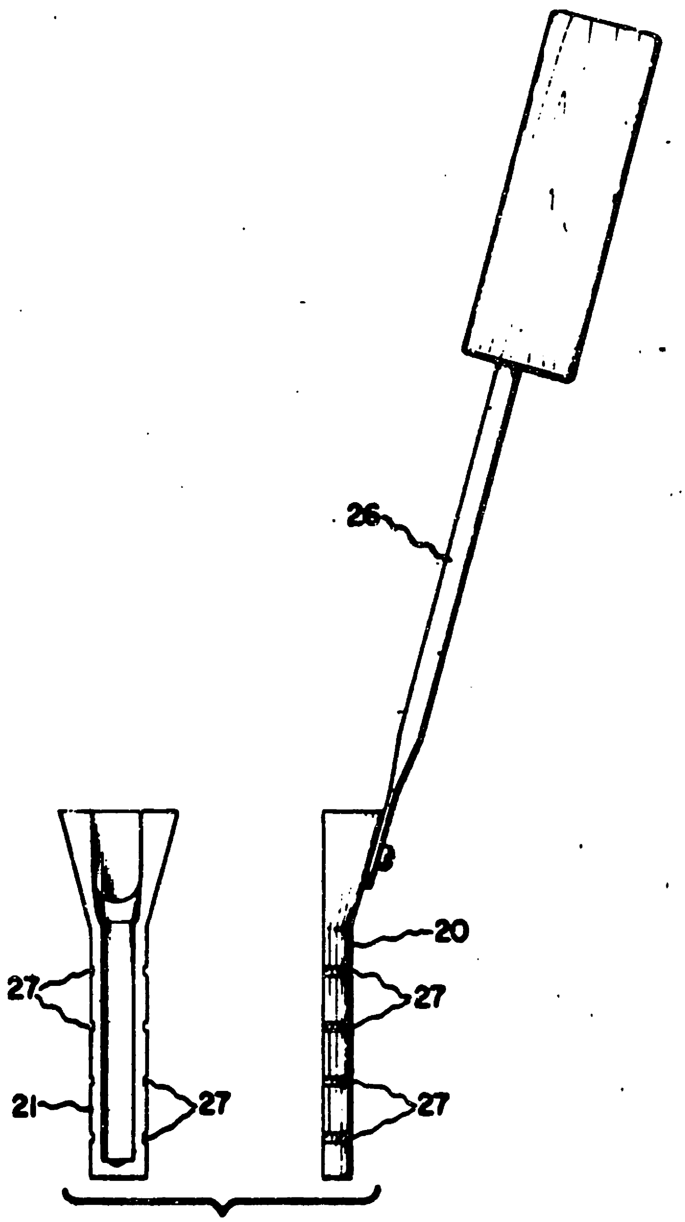


Fig. 2

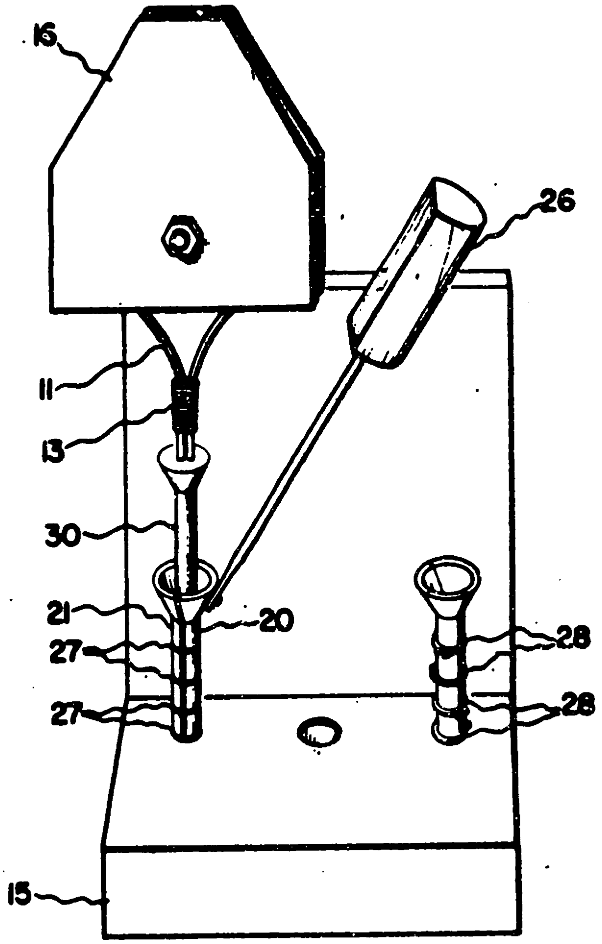


Fig. 4

# MOLD METHOD FOR SUPERCONDUCTIVE JOINT FABRICATION

## BACKGROUND OF THE INVENTION

The present invention relates to methods for fabricating superconductive joints, particularly in multifilamentary superconductive wires or cables. Even more particularly, the present invention relates to a mold based method for superconductive joint fabrication which employs agitation of the wires in a solution for stripping the metal matrix surrounding the multifilamentary superconductive strands.

A growing list of materials has now been found to exhibit superconductive properties when are cooled to temperatures below a critical value. Below this temperature all electrical resistance disappears. This permits the maintenance of current flow in superconductive circuits without external energy or power sources. In particular, superconductive conductors disposed in the form of electrical solenoids and coils of various configurations are capable of substantially continuous operation with no requirements to add additional electrical energy to the circuit. Superconductive circuits employing such solenoids are particularly advantageous in nuclear magnetic resonance (NMR) medical diagnostic imaging and spectroscopy systems. Moreover, superconductive circuits have found utility in a number of applications including power distribution and in magnetically levitated vehicles.

In any application in which superconductive wire is employed, it is almost variably necessary to employ one or more superconductive joints. However, to ensure that the resulting closed loop or circuit is entirely superconductive, it is necessary to ensure that the joint between the wire ends is also superconductive. However, superconductive materials display a tendency to undergo sudden and unexpected transitions to the resistive or ohmic state from the superconductive state. This phenomenon is referred to as quenching. The reasons for this phenomenon are not thoroughly understood. However, it is strongly believed that localized heating effects contribute to the phenomenon. However, the precise physical reasons for quenching do not yet appear to be fully understood. Accordingly, methods for its prevention are best describable as empirical rather than theoretical.

While quenching phenomena can occur in any portion of a superconductive circuit, it nonetheless appears that superconductive joints are in fact particularly susceptible to quench phenomena. Quenching is undesirable for at least three reasons. Firstly, quench conditions require restoration of the current in the circuit. Secondly, quench conditions often result in the undesired heating of the cryogenic fluid, typically liquid helium. Thirdly, quenching can cause damage to unprotected circuit elements. Accordingly, because of the undesired consequences of quenching and because of the particular susceptibility of superconductive joints to quench phenomena, it is seen that it is important to fabricate superconductive joints which are as immune as possible to this phenomenon.

In the case in which superconductive wires are to carry high levels of electrical current, for example 1,000 amperes and above, it is common practive to employ multifilamentary superconductive material. Typically such superconductive material comprises a carrier or matrix metal such as copper or copper-nickel alloy or a similar matrix conductor in which filaments of niobium-titanium alloy are incorporated. In such conductors an array of filaments are imbedded within a bulk carrier matrix. The formation of superconductive joints between multifilamentary wire ends poses particularly difficult problems. For example, in one form of superconductive joint the individual filaments are soldered to a superconductive sheet individually. This is a highly labor intensive operation. While this operation produces workable superconductive joints having even greater reliability against quenching.

A number of publications and patents deal with niobium-titanium superconductors and methods and means of forming joints between such superconductors.

One such publication is an article appearing in the October 1977 issue of *Welding Journal* starting at page 23 and titled "Soldering of Copper-Clad Niobium-Titanium Superconductor Composite" and dealing with the use of a variety of solders and fluxes. The solder joints were not superconducting. No flux was found which permitted and/or caused the solder to wet the superconducting filaments.

A method of forming a superconductive butt joint between copper-clad niobium-titanium superconductors by overwrapping the butt joint with smaller shunt superconductors and attaching the shunt in place by solder including a lead-bismuth solder is disclosed in U.S. Pat. No. 3,453,378. Various prior art methods of forming superconducting joints are disclosed in this patent as well as problems arising from failure of such joints.

The properties of various solders including solders containing lead and bismuth potentially useful in forming superconductive joints are disclosed in the article titled "Superconductivity Measurements In Solders Commonly Used for Low Temperature Research" appearing at page 180 of *Reviews of Scientific Instruments*, Vol. 40, January 1969.

A superconductive connection involving use of solders is described in U.S. Pat. No. 3,346,351 assigned to the same assignee as the present application.

A variety of superconductive solders and their uses are described in U.S. Pat. No. 3,156,539 also assigned to the same assignee as the subject application.

Formation of a superconductive joint employing combination of a superconductive low melting alloy containing combinations of lead-bismuth-tin and an outer crimped sleeve are taught in U.S. Pat. No. 3,449,818.

A method of making superconductive joints is also disclosed in U.S. Pat. No. 3,422,529 and is based upon the use of a crimped sleeve or cylinder which may comprise either stainless steel or a superconductive alloy. This patent however does not describe the use of solders or the multifilamentary condition and in particular requires twisting a pair of solid superconductive wires.

Accordingly, it is seen that many researchers have sought methods for forming reliable superconductive joints. It is also seen that the methods found have been sought empirically and that satisfactory explanations for the success of certain methods have not been forthcoming.

## SUMMARY OF THE INVENTION

In accordance with a preferred embodiment of the present invention a method for forming superconductive joints, particularly in multifilamentary superconductive wire, comprises a multistep process. First the ends of the multifilamentary superconductive wires to be joined are disposed in a hot liquid metal stripping bath for removal of the metal matrix. It is a particular feature of one embodiment of the present invention that these wire ends are moved or agitated relative to this stripping bath while disposed therein. The wire ends are then removed from the bath and inserted into a hot liquid superconductive solder bath. The wire ends are then removed from the solder bath, crimped if desired, and inserted into a mold. The mold, with wires in place, is then reinjected into the solder bath so as to fill the mold. The solder is allowed to solidify in said mold and the mold is removed from around said joint thereby forming a highly reliable superconductive joint. In practice, this process is preferably carried out in a controlled atmosphere, such as argon under standard temperature and pressure conditions. Accordingly, it is seen that the method of the present invention is capable of producing reliable superconductive joints and correspondingly reliable superconductive circuit elements such as coils, solenoids, windings and the like.

Accordingly, it is an object of the present invention to provide a reliable superconductive joint.

It is also an object of the present invention to provide a method which is particularly suitable for joining multifilamentary superconductive cables.

It is yet another object of the present invention to reduce the possibility of quenching in superconductive circuits.

It is also an object of the present invention to provide a method of superconductive joint fabrication which is not only repeatable but is also amenable to automation.

Lastly, but not limited hereto, it is an object of the present invention to provide reliable superconductive circuit elements.

## DESCRIPTION OF THE FIGURES

The subject matter which is regarded as the invention is particularly pointed out and distinctly claimed in the concluding portion of the specification. The invention, however, both as to organization and method of practice, together with further objects and advantages thereof, may best be understood by reference to the following description taken in connection with the accompanying drawings in which:

FIG. 1 illustrates a typical superconductive joint made by a prior art "dip and twist" process;

FIG. 2 illustrates mold halves employed in the present invention;

FIG. 3 illustrates a superconductive joint fabricated in accordance with the present invention;

FIG. 4 illustrates the molds and a joint of the present invention in position in a chill holder.

## DETAILED DESCRIPTION OF THE INVENTION

In a preferred embodiment of the present invention a superconductive joint between multifilamentary superconductive cables or wires is formed as follows. It is first necessary to remove any metal matrix surrounding the superconductive filaments from the end portions of the wires to be joined. This is accomplished by disposing the ends of the wires in a hot liquid metal stripping bath for removal of the metal matrix. Typically, the metal matrix comprises either copper or an alloy of copper and nickel. A suitable stripping bath comprises a material such as tin in which the copper or copper/nickel alloy is soluble. The tin stripping bath is typically maintained at a temperature of approximately 400° C. For a superconductive wire in which a copper matrix surrounds filaments of superconductive niobium-titanium, it is preferable to maintain the wires in such a stripping bath for approximately 45 minutes. In the case in which a copper-nickel matrix surrounds niobium-titanium superconductive strands, it is preferable to maintain the wire ends to be stripped in the tin bath for a period of approximately 180 minutes, again at a temperature of approximately 400° C. It is also important for the preferred embodiment of the present invention to mechanically agitate or move the wire ends relative to the fluid in the stripping bath. This provides a flushing action which facilitates complete removal of the surrounding matrix material. One means for accomplishing this flushing action is to rotate either the bath containing the wire ends or the mechanism holding the wire ends. For example, in a cylindrical bath having a diameter of approximately 6 inches, it has been found to be useful to rotate the bath at a rate of approximately 4 rotations per minute. Additionally, the tin employed in the stripping bath should be of high purity. For example, tin of "five-nines" purity in desirable, that is tin having a purity of 99.99999%.

Additionally, it is noted that it is preferable to carry out the various steps of the present invention in a controlled atmosphere. For example, the process may be carried out in a conventional laboratory "glove box" in which an inert atmosphere is present. An atmosphere comprising argon gas is preferred because of its inertness and relatively inexpensive cost. Xenon gas also could be employed, at atmospheric pressure as above, but the relatively higher cost of xenon gas makes argon the gas of preference. It is also noted that vacuum conditions also provide the kind of inert environment appropriate for carrying out the steps of the present invention. However, because of the relative difficulty of maintaining vacuum conditions such conditions do not comprise a preferred embodiment of the present invention. However, vacuum conditions may be more appropriate for commercial scale operations employing automated joint fabrication. It is also noted however that nitrogen does not in fact constitute a suitable atmosphere for the carrying out of the present invention. Additionally, it is preferred that the atmosphere in the glove box contained less than approximately 10 parts per million of moisture.

After an appropriate residence time in the stripping bath the wire ends to be joined are removed and disposed in a hot liquid superconductive solder, such as a mixture of lead and bismuth at a temperature of approximately 400° C. The lead bismuth solder which is preferred in the present method comprises approximately 40% lead and 60% bismuth by weight. However, a mixture containing approximately 35% lead and 65% bismuth or solutions in between these ranges are also employable. As in the tin stripping bath, high purity materials are desired. For example, it is preferred that the lead in the solder be 99.9999% pure and the bismuth to be 99.99999% pure. Additionally, the lead in the solder bath is preferably pretreated by deoxidation in a graphite mold for a period of one hour at a temperature of approximately 450° C. under vacuum conditions. In the case of niobium-titanium superconductive filaments disposed in a copper matrix a residence time of approximately 15 minutes in a lead-bismuth solder bath is preferred. In the case of niobium-titanium superconductive filaments disposed in a copper-nickel matrix it is desired to maintain the stripped ends of the wires to be joined in the superconductive solder bath for a period of approximately 45 minutes.

After an appropriate residence time in the solder bath the wire ends are removed, clustered or grouped together (if desired), inserted into a mold, and the ends are thereafter reinserted into the solder bath. Suitable molds are shown in FIGS. 2 and 4 which are more completely discussed below. After about 5 minutes in the solder bath the mold, now filled with solder, is removed and placed in a chill stand for cooling. Once the solder has solidified, the mold is removed thus releasing a solid, reliable superconductive joint which is typically between one and two inches in length. Additionally, it is also possible to impart a twist to the wire filaments following their removal from the superconductive solder bath and prior to their insertion into the mold.

For superconductive conditions existing at around 10° K. it is also possible to employ tin as a suitable superconductive solder. Accordingly, in such circumstances the solder bath and the stripping bath steps become merged.

FIG. 1 illustrates a prior art superconductive joint structure in which superconductive cable 11 in the shape of a wire loop is provided with superconductive joint 12. Wire loop portion 11 illustrates the condition of the wire prior to stripping of the copper matrix. The multifilamentary nature of the stripped cabling is evident in joint 12. For purposes of holding the wire ends together during joint formation, superconductive wire 13 is seen wrapped around the wires to be joined immediately above the joint. The joint illustrated in FIG. 1 is typical of the "dip and twist" structure described above.

FIG. 2 illustrates two mold halves employable in the process of the present invention. Mold halves 20 and 21 also preferably include grooves 27 for insertion of C-clamps for holding the mold halves together. However, any other convenient clamping mechanism can be employed. FIG. 2 also illustrates that one of the mold halves 20 is fitted with handle 26 for ease of manipulation particularly with respect to insertion into the hot solder bath.

FIG. 3 illustrates superconductive joint 30 fabricated in accordance with the present invention. In particular it is seen in this Figure that some superconductive wire already exists in the form of twisted niobium-titanium strands. It is therefore also seen that the process of the present invention is applicable to such conductors, and it is clear that for such conductors the matrix stripping operation is unnecessary and is accordinglyl by-passed.

FIG. 4 illustrates an empty mold with C-clips 28 holding halves 20 and 21 of the empty mold together in chill holder 15. Also illustrated is convenience holder 16 holding loop 11 of superconductive cable the ends of which have been provided with superconductive joint 30 which is seen being removed from mold halves 20 and 21.

From the above, it should be appreciated that the method of superconductive joint manufacture of the present invention provides a rigid and reliable superconductive joint which is significantly less prone to quench occurrences. It should also be appreciated that the method of the present invention is readily automatable, the use of the mold resulting in uniform and reliable superconductive joints whose manufacture does not depend upon the art or skill of manual assembly. Accordingly, the method of the present invention produces superconductive joints, solenoids, windings and general superconductive circuits which exhibit improved reliability and uniformity.

While the invention has been described in detail herein in accordance with the preferred embodiments thereof, many modifications and changes therein may be effected by those skilled in the art. Accordingly, it is intended by the appended claims to cover all such modifications and changes as fall within the true spirit and scope of the invention.

The invention claimed is:

1. A method for forming superconductive joints in multifilamentary superconductor wire embedded in a metal matrix, said method comprising the steps of:

disposing the ends of the multifilamentary superconductor wires to be joined in a hot liquid metal stripping bath for removal of said metal matrix;

removing said wire ends from said stripping bath;

disposing said wire ends in a hot liquid superconductive solder;

removing said wire ends from said hot liquid superconductive solder;

inserting said wire ends in a mold and filling said mold with hot liquid superconductive solder; and solidifying said solder.

2. The method of claim 1 further comprising the step of removing said mold.

3. The method of claim 1 in which said wire ends are moved relative to said stripping bath while disposed therein.

4. The method of claim 1 in which said multifilamentary wire ends are twisted prior to insertion into said mold.

5. The method of claim 1 in which said multifilamentary wire ends are grouped together prior to insertion into said mold.

6. The method of claim 1 in which said hot liquid stripping bath comprises tin.

7. The method of claim 6 in which said superconductive wire to be joined comprises niobium-titanium in a copper matrix and in which said wire ends are disposed in said stripping bath for a period of approximately 45 minutes.

8. The method of claim 6 in which said superconductive wire to be joined comprises niobium-titanium in a copper-nickel matrix and said wire ends are disposed within said stripping bath for a period of approximately 180 minutes.

9. The method of claim 1 in which said hot liquid superconductive solder comprises a mixture of lead and bismuth.

10. The method of claim 9 in which said bismuth is present in amounts ranging from approximately 35% to 40% by weight and said lead is present in amounts from approximately 65% to 60% by weight.

11. The method of claim 10 in which said superconductive wire to be joined comprises niobium titanium in a copper matrix and said wire ends are disposed in said solder bath for a period of approximately 15 minutes.

12. The method of claim 10 in which said superconductive wire to be joined comprises niobium-titanium in a copper-nickel matrix and in which said wire ends are disposed in said solder bath for a period of approximately 45 minutes.

13. The method of claim 1 in which moving said wire is performed by relative rotational movement of said wire ends in said stripping bath.

14. The method of claim 1 in which said process is carried out in a controlled atmosphere.

15. The method of claim 14 in which the atmosphere comprises gases selected from the group consisting of argon and xenon.

16. The method of claim 14 in which said process is carried out under vacuum conditions.

17. The method of claim 14 in which said atmosphere contains less than 10 parts per million of moisture.

18. The method of claim 1 in which said mold includes an elongate, substantially cylindrical cavity for insertion of said wire ends.

19. The method of claim 1 in which said mold comprises separate portions to facilitate removal of the resulting solidified superconductive joint.

20. The method of claim 1 in which said mold comprises material to which said solder does not wet.

21. The method of claim 1 in which said solder comprises tin.

22. The method of claim 1 in which said wire ends are removed from said solder bath prior to insertion of said wire ends in said mold.

23. The method of claim 1 further including the step of inserting said wire ends in said mold in said solder bath.

24. The superconductive joint produced in accordance with the method of claim 1.

25. The superconductive loop produced by the formation of a superconductive joint in accordance with the method of claim 1.

26. A method for forming superconductive joints between a plurality of strands of superconductive wire, said method comprising the steps of:

disposing the ends of the wires to be joined in a hot liquid metal superconductive solder;

removing said wire ends from said hot liquid superconductive solder;

inserting said wire ends in a mold with and filling said mold with hot liquid superconductive solder; and

solidifying said solder.

27. The superconductive joint produced in accordance with claim 26.

28. The method of claim 1 in which said mold includes a portion exhibiting a cross section having a plurality of straight sides, whereby the completed joint includes a portion for facilitating the immobilization of said joint.

29. The method of claim 26 in which said mold includes a portion exhibiting a cross section having a plurality of straight sides, whereby the completed joint includes a portion for facilitating the immobilization of said joint.

* * * * *

US Patent 4,901,429

**United States Patent** [19]

**Srivastava**

[11] **Patent Number:** **4,901,429**

[45] **Date of Patent:** **Feb. 20, 1990**

[54] **METHOD AND APPARATUS FOR MAKING A SUPERCONDUCTING JOINT**

[75] **Inventor:** **Vishnu C. Srivastava, Florence, S.C.**

[73] **Assignee:** **General Electric Company, Milwaukee, Wis.**

[21] **Appl. No.:** **157,346**

[22] **Filed:** **Feb. 17, 1988**

[51] **Int. Cl.⁴** .................................... **H01L 9/20**
[52] **U.S. Cl.** .................................... **29/599; 29/868**
[58] **Field of Search** .................... **29/599, 860, 868, 869**

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*Primary Examiner*—Howard N. Goldberg
*Assistant Examiner*—Carl J. Arbes
*Attorney, Agent, or Firm*—Quarles & Brady

[57] **ABSTRACT**

A method of forming a superconductive joint between superconductors made of filaments of superconducting material embedded in a matrix material utilizes two baths of molten metals. One of the baths of molten metal is used for etching the matrix material away from the superconducting filaments and is vibrated at an ultrasonic frequency. The other bath is also vibrated at an ultrasonic frequency and is used to mold the etched superconductors into a superconducting joint. Superconducting joints may thereby be made under ordinary atmospheric conditions between superconducting wires having similar and/or different matrix materials.

**8 Claims, 1 Drawing Sheet**

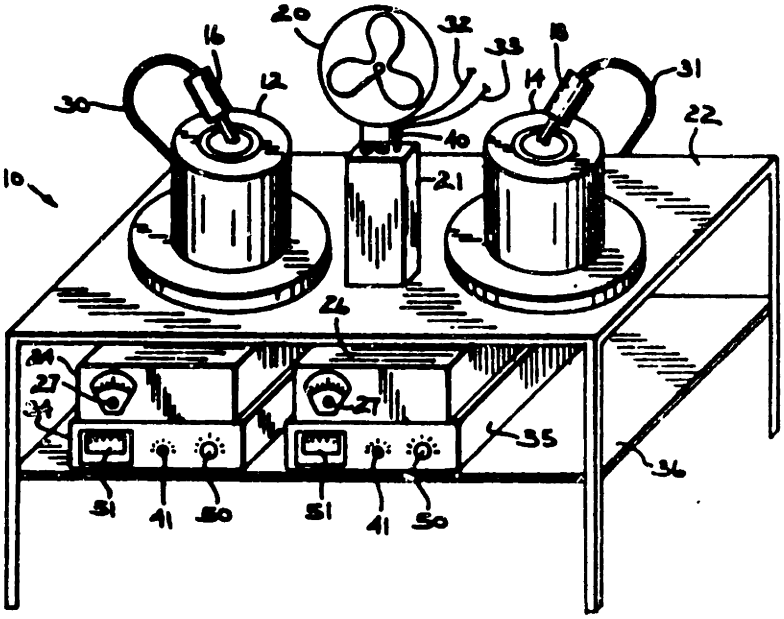


FIG. 1

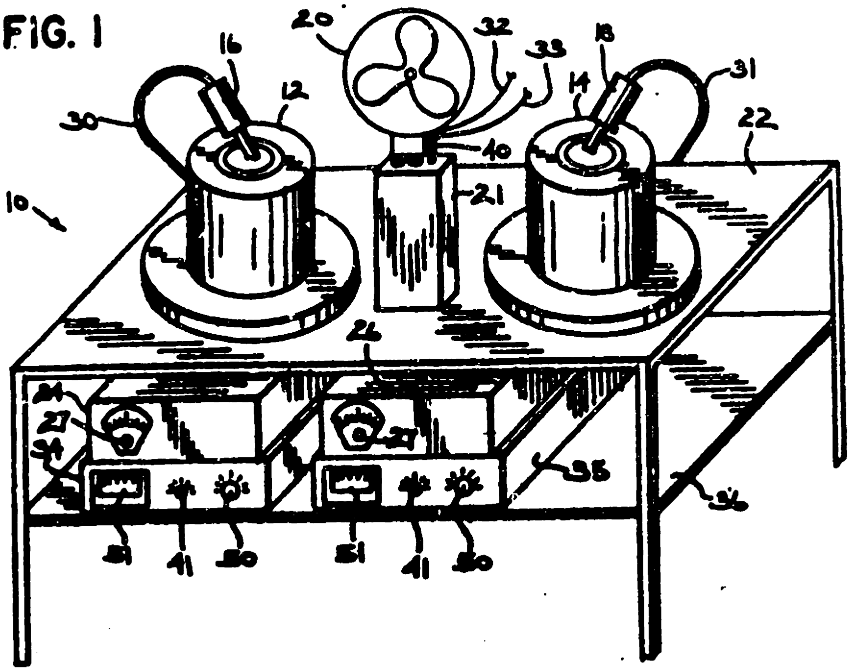
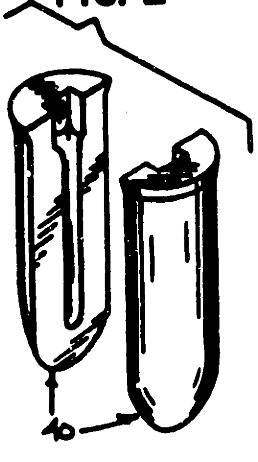


FIG. 2



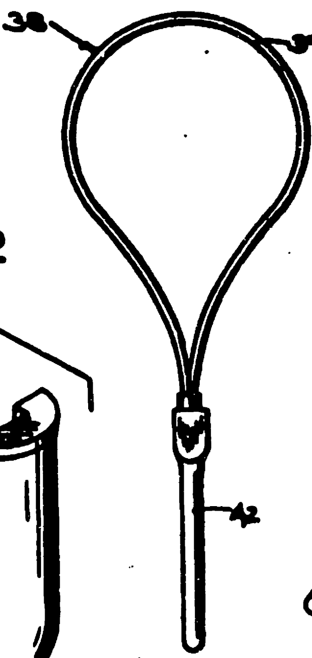


FIG. 3

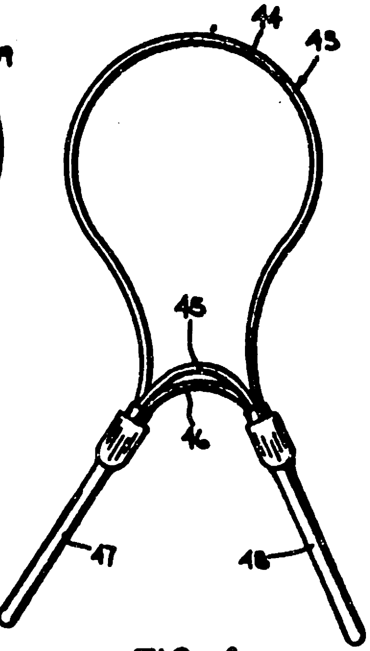


FIG. 4

# US Patent 4,901,429

## METHOD AND APPARATUS FOR MAKING A SUPERCONDUCTING JOINT

### BACKGROUND OF THE INVENTION

1. Field Of The Invention

This invention relates to a method and apparatus for making a superconducting joint and particularly to such a method and apparatus for making a superconducting joint between multifilamentary superconducting wires with similar and/or different matrix materials.

2. Discussion Of The Prior Art

It is well known that the resistance to current flow of many materials falls to zero, thereby making them superconducting, when they are cooled to a low temperature, known as the critical temperature. Superconducting wires typically comprise a superconducting material embedded in a matrix material, which may be, for example, copper or cupro-nickel, which is not itself a superconducting material. In the case of wire made of niobium-titanium alloy, which is a common superconducting material, the alloy may be in the form of filaments and the filaments encased by the matrix material.

It is necessary to form joints which are themselves superconducting to be able to usefully incorporate such superconducting wires into a superconducting circuit. For example, the wire may be wound into a coil to produce a high magnetic field, such as in an MR scanner. The coil cannot be made in a loop because it is necessary to be able to turn such a coil on and off. For that purpose, a superconducting switch is connected between the ends of the coil. To make the connection, it is necessary to make superconducting joints between the switch leads and the coil leads which result in a continuous circuit which is superconducting at the critical temperature and in a high magnetic field.

Methods are known for joining superconductors. One such method in disclosed in Thornton U.S. Pat. No. 4,584,547, which is owned by the assignee of the present invention. Thornton describes a method of making a superconducting joint which uses one or two pots of molten metal to etch the matrix material away from the filaments and then solder the filaments of the superconductors to be joined together. It is also known to use such a process to mold the filaments of the superconductors to be joined together with the molten metal while rotating each solder pot. This method is fully disclosed in Kumpitsch et al. U.S. Pat. No. 4,713,878, which is also owned by the assignee of the present invention. Typically in these processes, the etching metal used was tin and the soldering/molding material was lead-bismuth.

However, such processes were best performed in a controlled environment, such as a glove box, to insure producing good quality joints. Otherwise, oxides and other contaminants were believed to interfere with the joint and make it non-superconductive.

In addition, it has been difficult to join superconductors of one matrix material to superconductors of another matrix material, even if the superconductor material was the same. For example, copper matrix niobium-titanium multifilamentary wire was difficult to join to cupro-nickel matrix multifilamentary wire.

### SUMMARY OF THE INVENTION

The invention provides a method of making a joint between superconductors made of filaments of superconducting material embedded in a matrix material which overcomes the above disadvantages. As a part of the method, a bath of molten metal is provided and is vibrated at an ultrasonic frequency. This vibrating bath is then used to etch and/or mold the ends of one or more superconductors into a superconducting joint. In this way, a highly reliable superconducting joint capable of conducting high currents is produced. The method may be performed under ordinary atmospheric conditions and may also be used to make joints between superconductors having different matrix materials.

In a preferred form, a bath of one molten metal is used to etch the superconductors to be joined and a bath of another molten metal, which is a superconducting material, is used to mold the joint. Both baths are vibrated at an ultrasonic frequency, the first bath being vibrated to remove the matrix material from the ends of the superconductors and the second bath being vibrated to remove the metal of the first bath and further wet the ends. A mold is then filled with molten metal from the second bath and the ends of the superconductor are placed in the mold and solidified therein to form the joint.

In the preferred form, ultrasonic horns are used to vibrate the molten metal baths. A horn is placed near the center of each bath with its tip submerged therein. The ends of the superconductors to be etched and/or molded are then placed in the baths close to the horn.

It is therefore a principal object of the invention to provide a reliable method of making superconducting joints.

It is another object of the invention to provide such a method which may be easily performed under atmospheric conditions.

It is another object of the invention to provide such a method which may be used to join superconductors which are made of different matrix materials.

It is another object of the invention to provide such a method for making superconducting joints which are capable of carrying a high persistent current in a magnetic field.

These and other objects of the invention will be apparent from the drawings and from the detailed description.

### DESCRIPTION OF THE DRAWINGS

The present invention is described below, as required by 35 U.S.C. §112, in such full detail as to enable those skilled in the art to practice the invention and also to set forth the presently-contemplated best mode for its practice, all by reference to the following drawings in which:

FIG. 1 is a perspective view of an apparatus for performing the method of making a superconducting joint of the present invention;

FIG. 2 is a perspective view of a mold for making a superconducting joint of the present invention;

FIG. 3 is an elevation view of a superconducting loop made with a joint of the present invention; and

FIG. 4 is an elevation view of another superconducting loop made with two joints of the present invention.

### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to FIG. 1, an apparatus 10 for performing a method of making a superconducting joint according to the invention is disclosed. The apparatus shown includes two heated pots 12 and 14 for melting metals placed therein and holding them at a set temperature, ultrasonic horns 16 and 18 in the pots 12 and 14, respectively, and a fan 20 and phenolic holding fixture 21 for cooling completed joints, all supported on a table 22.

The pots 12 and 14 are standard solder pots and are controlled respectively by power supplies 24 and 26. Each power supply 24 and 26 has a dial 27 with which the temperature of the corresponding pot can be set. The range of temperatures in the preferred embodiment which can be set is 0°-600° C. and the set temperature is accurate to within 2° C. Generally, the normal range in which the temperature will be set for making joints of the invention is 100°-500° C. In the preferred embodiment, the inside dimensions of each of the pots 12 and 14 is five inches deep and five inches in diameter.

Each horn 16 and 18 has a cord 30 and 31 which is connected to a controller 34 and 35, respectively, supported on a shelf 36 of the table 22. Each horn 16 and 18 may be of the piezoelectric crystal transducer type and operate at a frequency of approximately 20 kilohertz. In the preferred embodiment, each horn 16 and 18 has a maximum acoustical output power of approximately 150 watts. The generators 34 and 35 are matched to the horns 16 and 18 and are usually initially purchased as a unit. Horns found suitable in the preferred embodiment are the Model BP-4 and generators found suitable are the Model SG-250, both of which are commercially available from Blackstone Corporation, Ultrasonic Division, Jamestown, NY 14701. The horns 16 and 18 are 2¼" in diameter by 11" long with a 1⅛" diameter aluminum output coupling piece.

The pot 12 is filled with molten tin (Sn) and the pot 14 is filled with molten lead-bismuth (Pb-Bi). The tin is 99.99% pure and the lead-bismuth is a 40% lead, 60% bismuth (by weight) alloy with the lead and bismuth going into the alloy each being 99.999% pure and the maximum impurities in the alloy being 95 parts per million. The temperature of the molten tin and of the molten lead-bismuth varies according to the particular types of wire being joined, but usually is in the range of 300°-450° C.

The horns 16 and 18 must be properly coupled to the molten metals in the pots 12 and 14. Each horn should be oriented at an angle of about 45° and positioned with its end entering approximately the center of the corresponding pot. The tip of the each horn should be submerged in the molten metal at least 1 inch.

The horns are turned on and allowed to stabilize within the pots. It may also be necessary to provide a cooling air flow, such as from a compressed air source, to cool the ultrasonic probes, according to the manufacturer's specifications. The horns also must be tuned after they are on, and this usually requires setting a power level control 50 and then adjusting a tuner control 41 to a null, where a power meter 51 indicates the lowest reading at the power setting, of each of the controllers 34 and 35. After being tuned, the power meter 51 is reset to zero, and the system is allowed to stabilize for 10-15 minutes. Then, the power meter 51 is adjusted to a reading of 50-75 watts and the apparatus is ready to begin making joints. However, as the horns are operating in the pots 12 and 14, they erode so that during the course of production, the power readings will change and tuning will be necessary periodically. When the horns can no longer be tuned to their lowest power reading, they must be replaced.

The general procedure for molding joints according to the invention begins with assuring that both pots have stabilized at the appropriate temperature for the particular type of joint to be made. A running log of the number and type of joints made is also kept for each of the pots to assure that the pot has not reached an unacceptable level of impurities. The number of joints allowed before changing the metals in the pots will depend upon the size of the wires and the size of the pots. Further discussion may be found in the examples which follow.

If the wires are insulated, the insulation, which may be made of, for example, formvar varnish, is then stripped from the ends of the wires to be joined. Approximately 2" from the ends, the wires to be joined are fastened together using a piece of thin copper wire and a shallow mark is scribed into the wires approximately 1.5" from the ends. Oxides and other impurities which have formed are then skimmed from the top of the tin pot 12 and the wires are inserted into the pot up to the 1.5" scribe mark. The wires are inserted near the horn blade but not touching it. The wires are held in the pot 12 for a certain time, which also varies according to the type of joint being made. The wires may be held manually, but preferably are held with a suitable clamping fixture (not shown).

Placing the wires in the tin in the pot 12 etches the matrix material away from the filaments of the superconducting wires. This must be done to electrically couple the wires to be joined. The matrix material disperses into the tin bath under the action of the ultrasonic horns and it is believed that such action contributes to high purity tin being in the immediate vicinity of the wires in the center of the pot near the horn. It is also believed that the ultrasonic energy imparted to the tin bath helps to more completely remove the matrix material from the filaments, thereby helping to reduce the amount of impurities in the final joint.

After the time for etching in the tin has expired, the impurities are skimmed from the top of the tin and from the top of the lead-bismuth (such as with stainless steel spoons, not shown) and the wire ends are transferred from the tin to the lead-bismuth in the pot 14. In the lead-bismuth, the wires are also positioned adjacent to but not touching the horn 18 and up to the 1.5" scribe mark. Under the action of the ultrasonic horn 18 in the lead-bismuth pot 14, the tin is removed from the filaments as well as any remaining matrix material or other impurities.

The temperature and dipping time in the lead-bismuth depends upon the particular type of joint being formed and the wires used. The wires are in the lead-bismuth for a certain set time, during which a two-part mold (FIG. 2), which is fastened together such as with appropriate clips (not shown), is placed in the lead-bismuth. The mold 40 is allowed to fill with the lead-bismuth and when the set time in the lead-bismuth for the joint has expired, the mold is emptied, the wires are placed in the mold, and the mold is then refilled with lead-bismuth. During this operation, impurities forming on top of the lead-bismuth should be removed.

After the mold 40 is filled, it is tapped to remove any air bubbles and checked to assure that the filaments of the wires are completely inside the mold. If the mold 40 leaks, it is refilled with lead-bismuth. The mold, with wires 32 and 33 therein, is then placed in the fixture 21 in front of the fan 20 to cool as shown in FIG. 1. Appropriate fixtures (not shown) may be used to hold the mold 40 and the wires 32 and 33 in the pot 14 and to hold the wires in the molten lead-bismuth when the mold is in the fixture 21. When the lead-bismuth has solidified, the mold is removed, along with any excess flashing, to produce a finished joint.

## EXAMPLE 1

Joints were made according to the invention using a single section of copper-matrix wire, hereby designated wire W, and joining its ends together. The superconducting wire W is a copper matrix niobium-titanium multifilamentary superconducting composite conductor. Preferably, the superconducting material of the filaments is 46.5 plus or minus 1.5 weight percent titanium. The matrix material is CDA-10100 (oxygen free electronic) copper. The volume ratio of copper to niobium-titanium is a minimum of 2.3:1 in the final conductor. Some of the wire used to make joints according to this example was also insulated with a formvar varnish coating.

The dimensions of each wire W prior to insulation is preferably 0.050 by 0.100 inches nominally. If insulated, the insulated wire dimensions are nominally 0.053 by 0.103 inches and has a minimum 0.025 inch flat on the 0.053 inch side and a minimum 0.075 inch flat on the 0.103 inch side. The equivalent average filament size in the final conductor is preferably between 30 and 60 microns in diameter. The twist pitch length of the wire should be 3.0 plus or minus 0.5 inches. As such, the minimum critical current of this wire should be 3200 amps in a magnetic field of 2 Tesla at 4.2° K. and 2000 amps in a magnetic field of 4 Tesla at 4.2° K.

To make a joint with the wire W, the metals in both pots 12 and 14 were held at 400° C. The etching time in the tin was 15-18 minutes and the soaking time in the lead-bismuth was 8-10 minutes. Eight of these joints were made using the single loop construction 38 shown in FIG. 3, which includes a wire 39 and a joint 42. These eight joints were tested and found capable of conducting persistent currents (i.e., equivalent decays of less than 0.1 ppm/hr.) ranging from 2820-3180 amperes.

Starting with new tin and new lead-bismuth, approximately 60 of the joints 42 of this example could reliably be made using the apparatus of the preferred embodiment before replacing the metals. Also, in production, it was found desirable (for a margin of safety) to reduce the temperature of the metal baths to 350° C., increase the etching time in the tin to 30 minutes, and adjust the soaking time in the lead-bismuth to 7-10 minutes, with comparable current carrying results being obtained.

## EXAMPLE 2

Other joints were made according to the invention in which a single length of the wire W, described above, was joined in parallel to two lengths of a wire hereby designated as X. The superconducting wire X is a cupro-nickel matrix niobium-titanium multifilamentary (576 filaments) superconducting composite conductor. Preferably, the superconducting material of the filaments is 46.5 plus or minus 1.5 weight percent titanium. The matrix material is 70% copper, 30% nickel with a nominal resistivity of $3.2\times10^{-5}$ ohm-cm at room temperature. The outer jacket of the wire is 90% copper, 10% nickel with a nominal resistivity of $1.5\times10^{-5}$ ohm-cm at room temperature. The volume ratio of cupro-nickel to niobium-titanium is nominally 1.10:1 in the final conductor. Each wire is also fully cured formvar varnish coated for insulation.

The diameter of each wire X prior to insulation is preferably 0.041 inches nominally. The insulated wire diameter is nominally 0.044 inches. The equivalent average filament size in the final conductor is preferably 29 microns in diameter nominally and should not be greater than about 32 microns. The twist pitch length of the wire should be 1.0 plus or minus 0.25 inches. As such, the minimum critical current of this wire should be 630 amps in a magnetic field of 5 Tesla at 4.2° K.

A finished test loop 43 including wires 44, 45 and 46, and joints 47 and 48, as made in this example is shown in FIG. 4. Five of the test loops 43 were made in each of two batches. In the first batch, both metals in the pots 12 and 14 were held at 400° C., the etching time in the tin was 25-30 minutes, and the soaking time in the lead-bismuth was 8-10 minutes. In the second batch, both metals in the pots 12 and 14 were held at 450° C., the etching time in the tin was 15-18 minutes, and the soaking time in the lead-bismuth was 8-10 minutes. Loops from the first batch exhibited persistent current carrying capacities ranging from 1978-2087 amperes and loops from the second batch attained persistent currents in the range of 1456-1864 amperes. The quench currents for the first batch were also measured and found to be 3362-4247 amperes.

In production, the temperature and time parameters found most desirable were: temperature of both baths: 400° C., etching time in tin: 25 minutes, soaking time in lead-bismuth: 7-10 minutes. Starting with new tin and new lead-bismuth, 180 or more of these joints could reliably be made using the apparatus of the preferred embodiment before finding it desirable to change the metals in the pots 12 and 14.

## EXAMPLE 3

Other loop configurations like that shown in FIG. 3 having only one joint of the invention were made with three strands of the wire X (instead of only one strand as shown in FIG. 3). In making these test loops, both baths were held at 400° C., the etching time in the tin was 40-60 minutes, and the soaking time in the lead-bismuth was 7-10 minutes. Five of these loops were made and were found to conduct persistent currents ranging from 1500-2227 amperes and quench currents ranging from 3500-4626 amperes.

## EXAMPLE 4

Other parallel circuit configurations like that shown in FIG. 4, but with three wires between the joints 47 and 48 instead of the two wires 45 and 46, were made having joints according to the invention. In these test loops, the three lengths were of wire X and the one length was of wire W. Both bath temperatures were approximately 400° C., the etching time in the tin was 40-60 minutes, and the soaking time in the lead-bismuth was 7-10 minutes. Persistent current measurements for five of these joints fell in the range 1597-2551 amperes and quench currents were measured to be in the range 3383-5201 amperes.

## EXAMPLE 5

Other parallel circuit configurations like that shown in FIG. 4 (but with only one wire between the joints 47 and 48 instead of the two wires 45 and 46) were made having joints according to the invention. In these test loops, one length of wire hereby designated Y and one length of the wire X were joined with two joints of the invention.

The superconducting wire Y is a copper matrix niobium-titanium multifilamentary superconducting composite conductor. Preferably, the superconducting material of the filaments is 46.5 plus or minus 1.5 weight percent titanium. The matrix material is CDA-10100 (oxygen free electronic) copper. The volume ratio of copper to niobium-titanium is 2.0 (±0.15):1 in the final conductor. Each wire is also fully cured formvar varnish coated for insulation.

The diameter of the wire Y prior to insulation is preferably 0.0273 inches nominally. The insulated wire diameter is nominally 0.0293 inches. The equivalent average filament size in the final conductor is nominally 40 microns in diameter. The twist pitch length of the wire should be 1.25 plus or minus 0.25 inches. As such, the minimum critical current of this wire should be 500 amps in a magnetic field of 1 Tesla at 4.2° K. and 275 amps in a magnetic field of 3 Tesla at 4.2° K.

For these joints, the pots 12 and 14 were optimally held at 400° C., the wires were etched in the tin for 15-18 minutes, and soaked in the lead-bismuth for 7-10 minutes. Twenty of these joints were made and exhibited a persistent current capacity falling in the range of 474-695 amperes and quench currents in the range of 732-862 amperes.

Five more joints were made as in this example but with the temperature of both baths 450° C., the etching time in the tin 12-15 minutes, and the soaking time in the lead-bismuth 7-10 minutes. These joints exhibited persistent current capacities in the range of 448-568 amperes.

All of the above joints were made under atmospheric conditions without any protective shielding or gases. Moreover, the joints made as described in examples 2, 4 and 5, above, were between superconductors having different matrix materials (wires W and Y have copper matrices and wire X has a cupro-nickel matrix). In addition, as illustrated by examples 1 and 3, above, the method is also effective to join superconductors of the same matrix material, and does so whether the matrix material is copper or an alloy, such as cupro-nickel.

Many modifications and variations of the preferred embodiment will be apparent to those of ordinary skill in the art but will still be within the spirit and scope of the invention. For example, metals other than tin and lead-bismuth could be used as long as the metal in the pot 12 is a solvent of the superconducting wire matrix material and the metal in the pot 14 is a superconductive solder. Also, it may be possible in some applications to use a single molten bath to do the etching and the molding. Therefore, the invention should not be limited by the scope of the preferred embodiment, but only by the claims which follow.

I claim:

1. A method of making a joint between superconductors made of filaments of superconducting material embedded in a matrix material including the steps of:

stripping the matrix material away from ends of the superconductors to be joined;

providing a heated soldering bath of molten metal, said metal being a superconducting material;

vibrating the soldering bath at an ultrasonic frequency;

submerging the stripped ends to be joined of the superconductors into the vibrating soldering bath;

filling a mold with the superconductive material of the soldering bath in a molten state;

placing the ends of the superconductors to be joined into the molten superconductive material in the mold; and

cooling the superconductive material in the mold to encase the ends of the superconductors in the solidified superconducting material of the soldering bath.

2. A method as in claim 1, wherein the matrix materials of the superconductors to be joined differ.

3. A method as in claim 1, wherein the method is performed under atmospheric conditions.

4. A method as in claim 1, wherein the bath is vibrated by an ultrasonic horn at least partially submerged in the bath.

5. A method as in claim 4, wherein the horn is positioned approximately in the center of the bath.

6. A method as in claim 4, wherein the end of the superconductor is dipped in the bath adjacent to the horn.

7. A method as in claim 1, wherein the step of stripping the matrix material away from ends of the superconductors to be joined comprises:

providing an etching bath of molten metal for stripping the matrix material away from the ends of the superconductors to be joined;

vibrating the etching bath at an ultrasonic frequency; and

holding the ends of the superconductors to be joined in the etching bath until the matrix material is stripped from the filaments.

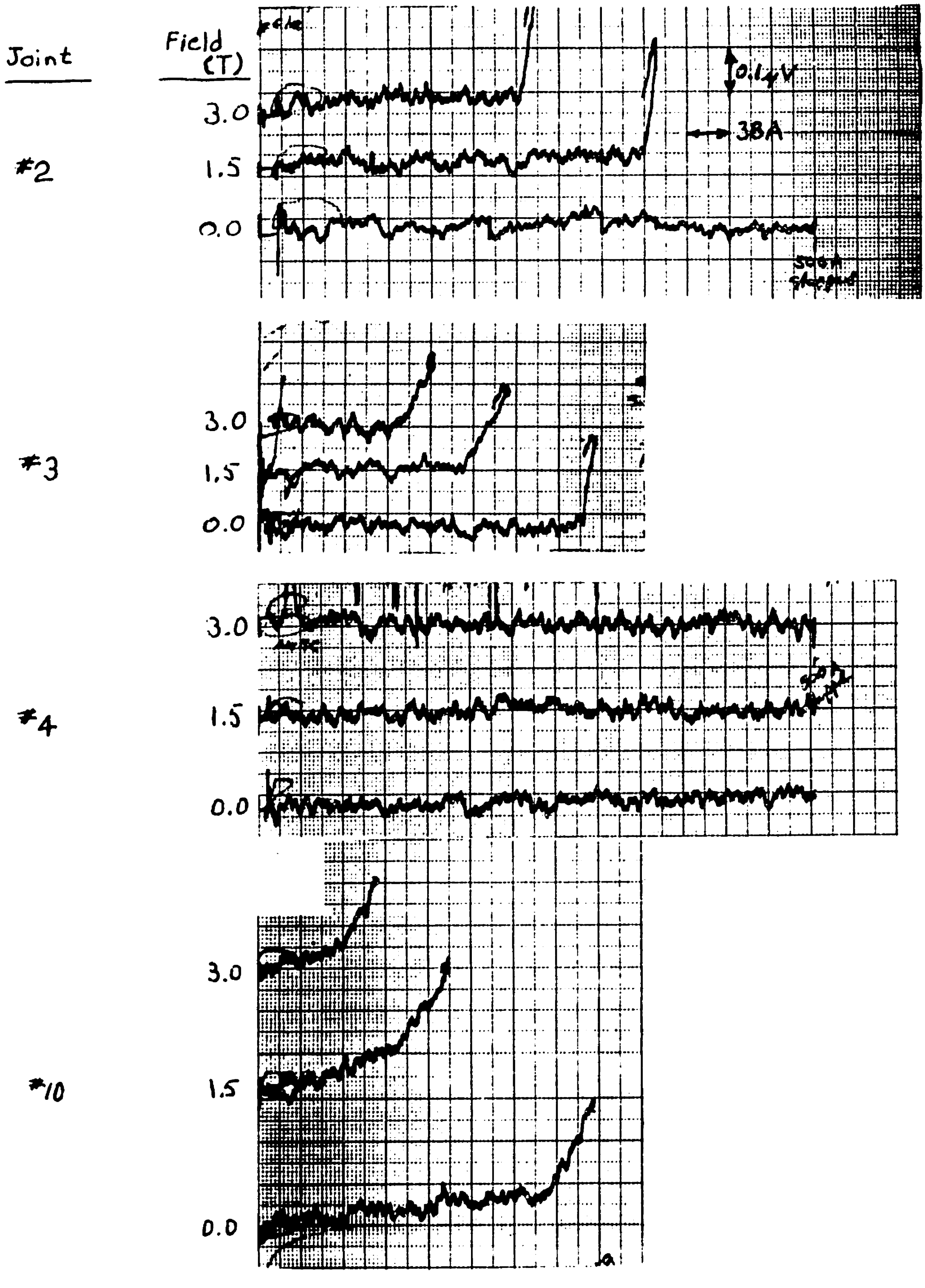
8. A method as in claim 7, wherein the soldering and etching baths are two different baths and are of different metals.

* * * * *

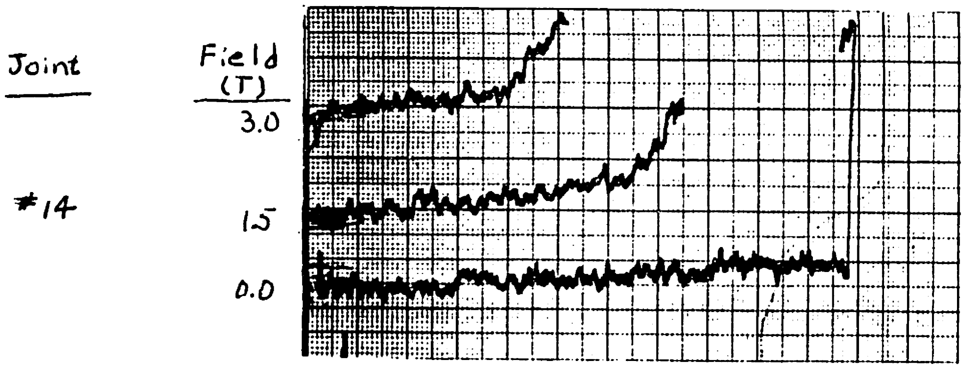
# Appendix B

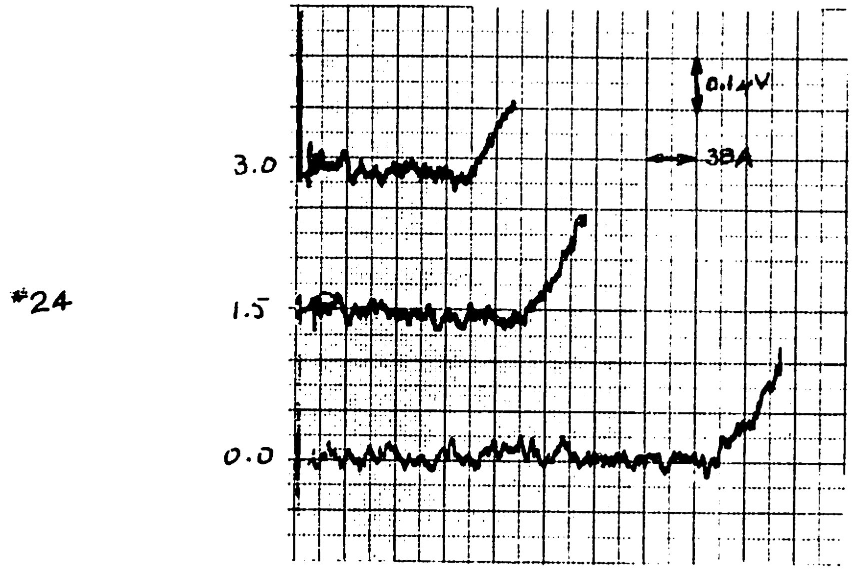
This appendix includes copies of the raw data for selected joints. The V–I traces were copied directly from data taken for performance and reliability tests. The plots have been slightly reduced in size so the scale is included on each page.

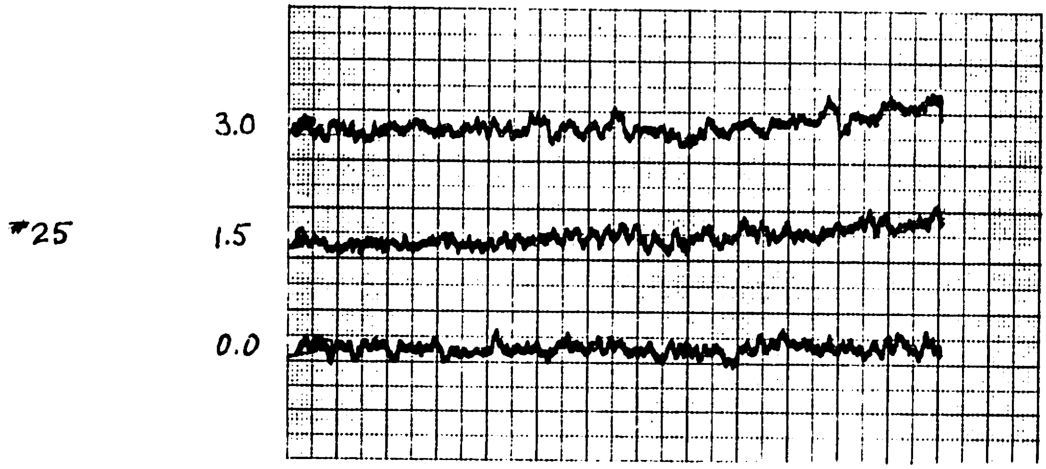
# Performance Test Data



## Performance Test Data







## Performance Test Data