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Taeyoung Pyon for the degree of Master of Science in Mechanical Engineering presented on November 9, 1992.

Title: Evaluation of Copper to Superconductor Area Ratio Measurement Techniques for Niobium-Titanium Composite Superconducting Wire

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William H. Warnes

Measurement of the copper to superconductor ratio (Cu/SC) in composite superconducting wires is necessary for process control, quality assurance, and characterization of the final wire properties.

A comparison has been made of three techniques for measuring the Cu/SC ratio on a set of commercial wires produced for the Superconducting Super Collider (SSC) Laboratory.

The simplest and most straightforward technique, chemical etching, was found to display the best reproducibility, while the electrical resistivity technique shows the most variation and sensitivity to measurement errors, as well as being the most difficult to perform.

The image analysis technique is fast and fairly reproducible, and is capable of providing much more information on the wire parameters than either of the other techniques.

EVALUATION OF COPPER TO SUPERCONDUCTOR AREA RATIO MEASUREMENT TECHNIQUES FOR NIOBIUM-TITANIUM COMPOSITE SUPERCONDUCTING WIRE

by

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CHAPTER 1 INTRODUCTION

Since the discovery of superconductivity in the early 1900s, a number of metallic elements, alloys, and compounds have been found to become superconducting at low temperature. In order for the superconducting state to be acquired, one must lower the temperature below a critical temperature (T_c), usually near absolute zero. By doing so, the primary physical property of superconductivity, that is zero electrical resistance, can be obtained.

For most superconducting materials and applications, it is desirable to make wires as a composite. In other words, the superconductor needs to have a parallel path of normally conducting material, usually copper, which helps to carry currents and to diffuse heat to the coolant. Copper has been used as a successful stabilizing material because of its high electrical and thermal conductivity, high heat capacity, ductility, and good mechanical strength at cryogenic temperatures.

Another important aspect of fabricating superconducting wires is using a multifilamentary process rather than having a monofilamentary conductor. In order to keep electrical and thermal stability through the superconducting wire, the heat generated by magnetic flux motion in the superconducting state must be removed to the coolant. Because the thermal diffusion time of a superconducting material is much bigger than that of copper, it is necessary to reduce the thermal diffusion time by breaking the superconducting material into small diameter filaments less than approximately 50μm [Ref 1].

Nb-Ti multifilamentary superconducting conductors, which are usually manufactured by hot extrusion followed by cold drawing, consist of fine superconducting filaments embedded in a copper matrix. The major stages of a process route for the fabrication of Nb-Ti multifilamentary composite wire are shown in Figure 1. It is most desirable for the magnet designer as well as for the conductor manufacturer to control the electrical and mechanical properties of conductors.

The geometry of the superconducting wire cross section is one of the critical aspects of composite design, for which the important geometric parameters, such as the overall and interfilamentary copper to superconductor area ratio (Cu/SC), Nb-Ti filament diameter (D), and spacing between the filaments (S) must be determined.

The filament diameter must be kept small not only for thermal stability concerns, but also to reduce magnetization effects, and filament spacing must be kept small enough to maintain a regular filament array and reduce sausaging of the filaments. Too small a spacing, however, leads to filament coupling which is undesirable because it causes flux jumping.

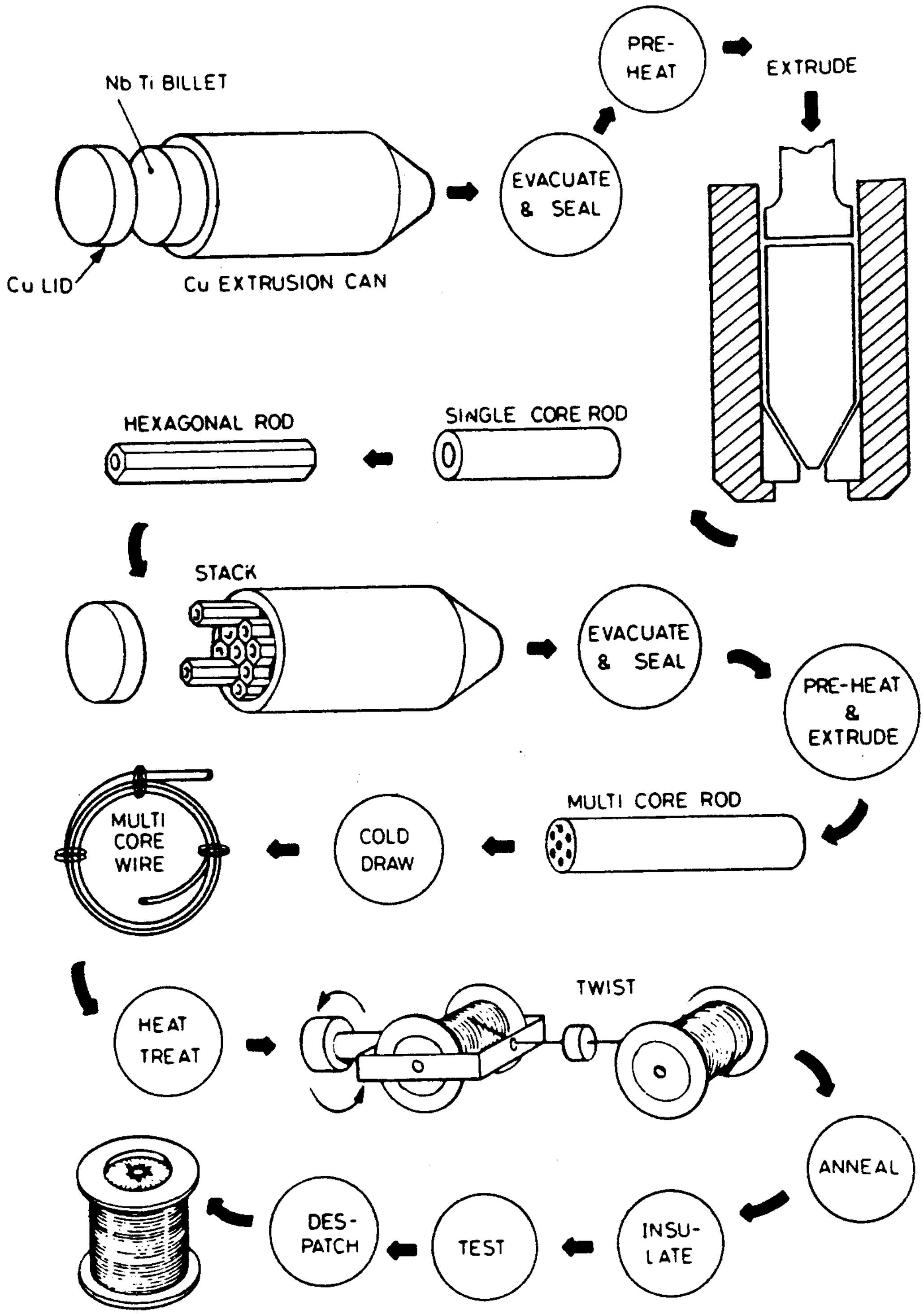


Figure 1. A schematic illustration of manufacturing process for Nb-Ti/Cu composite superconducting wire [Ref 1].

In order to produce a very small filament diameter, a lot of cold work is required which can possibly lead to mechanical instabilities such as filament sausaging and excessive wire breakage, eventually reducing the overall yield of wire.

For these reasons, the Cu/SC ratio needs to be controlled to assure the wire stability, both electrically and mechanically. Since the connection between these geometric parameters and wire performance and manufacturability has not been clearly understood, it is necessary to measure the Cu/SC ratio from a basic research point of view. Further, if the critical current density (J_c) remains constant, any variations in Cu/SC ratio imply variations in critical current (I_c) and this may result in reduced performance of the wire. In this respect the Cu/SC ratio can be esteemed as a very useful quality assurance tool for the composite superconducting wires.

1.1 Definition of Cu/SC Ratio

Knowing the respective cross section areas of the components of the composite superconducting wires has been a good way to interpret the behavior of the composite during the various manufacturing processes of the superconducting strand. Figure 2 shows the cross section of several commercial Nb-Ti composite wires. The fine filaments of superconductor are embedded in a copper matrix.

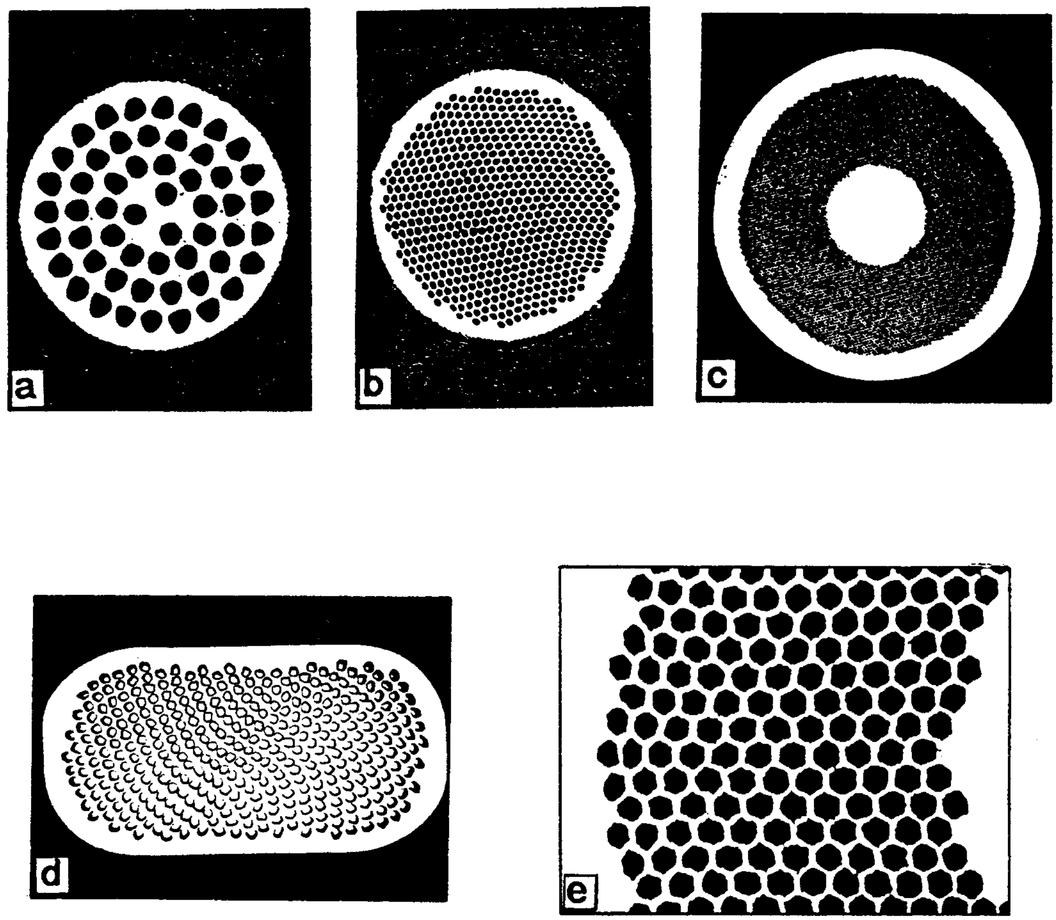


Figure 2. Cross section of several Nb-Ti/Cu composite superconducting wires. (a) 54-filament composite. (b) 583-filament composite. (c) 7248-fine filament composite wire used for SSC dipole magnet. (d) 361-filament composite rolled to a rectangular shape. (e) Partial cross section of a multifilamentary composite assembled using hexagonal monofilaments.

In filamentary composites, generally one or more sheets of niobium or vanadium foil is wrapped around the Nb-Ti filament in order to keep the reactive material from contaminating the high-purity stabilizer material and vice versa. This material is called the diffusion barrier which is therefore interposed between the filament and the copper matrix.

The Cu/SC ratio is defined as the area ratio of non-superconducting material to superconducting material in cross section of the superconducting wire. This ratio is very useful for the user as well as for the manufacturer to predict the critical current all along the composite, because the I_c directly depends on the superconductor cross section area. In other words, the critical current decreases as the amount of copper increases while the critical current density remains constant along the wire under uniformity of the strand. Figure 3 shows a correlation between I_c and Cu/SC ratio for a wide variety of Superconducting Super Collider (SSC) wires. This relationship may be useful to improve the manufacturing process and Cu/SC ratio control for the composite wires. The solid line running through the data is a linear least squares fit [Ref 3].

There are basically three reasons to measure Cu/SC ratio of the superconducting wire. This ratio is needed to determine J_c for a wire from the basic I_c measurement. A second reason is to assess the uniformity of the manufacturing process since I_c is proportional to the Cu/SC ratio as described above. Finally it is necessary to know the amount of copper in the cross section to define the safety margin of the magnet in the case of a quench.

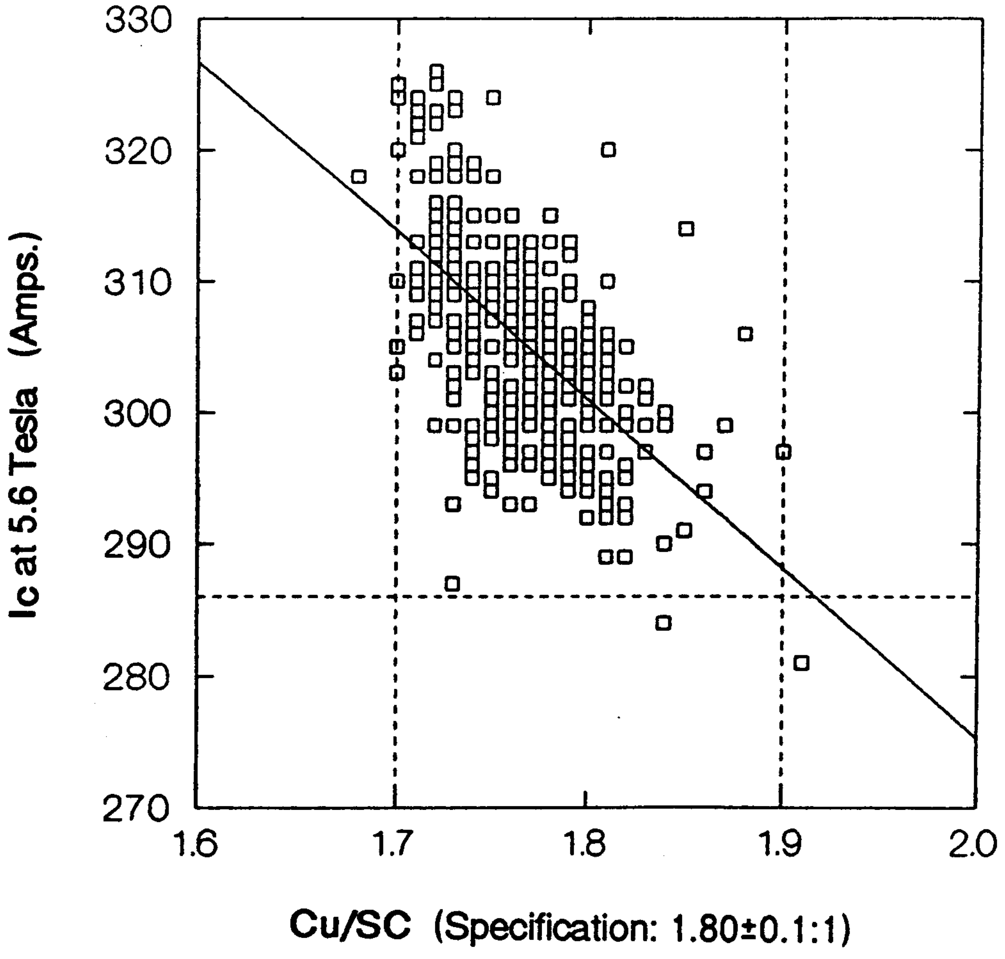


Figure 3. A plot of Ic versus Cu/SC ratio for a number of SSC outer strands produced by different manufacturers [Ref 2].

1.2 Nb-Ti Superconducting Wire For SSC Magnets

At present, the state-of-the-art in industrial fabrication of Nb-Ti superconductors is based on the wires manufactured for use in the SSC. The wires to be used in SSC magnets are routinely fabricated as multifilamentary composites consisting of Nb-Ti filaments in a high-purity and oxygen-free copper matrix. The superconductor composition is in the range of Nb47±1wt.%Ti, and is high homogeneity grade or equivalent. For the SSC the goal has been to increase the J_c of the wire so that, for the same I_c, the total amount of superconductor can be reduced which leads to less production cost of the wire. The specifications for SSC wire set the Cu/SC ratio at 1.80±0.10 for the outer wire, and 1.50±0.10 for the inner wire. The wires studied here are from an older SSC design inner wire for which the Cu/SC ratio is 1.30±0.10. The niobium barrier, at monofilament assembly size, consists of an area equal to a minimum of 4% of the Nb-Ti filament area. Measurement of the Cu/SC ratio has been in the past determined mainly by the chemical etching method because of its simplicity and reproducibility. Use of the electrical resistivity technique [Ref 3], however, is lately being pursued by the SSC Laboratory because of the potentially non-destructive aspect of this method. Metallographic polishing, coupled with image analysis, has recently provided a third method for Cu/SC ratio measurement. This work has been performed to establish the reproducibility among these three techniques for measuring the Cu/SC ratio on a set of commercial wires produced for the SSC magnets.

CHAPTER 2 EXPERIMENTAL TECHNIQUES

2.1 Description of Overall Experiments

The Cu/SC ratio measurements were performed on a set of SSC samples using three different techniques including chemical etching, electrical resistance, and metallographic image analysis. These were then compared to evaluate each experimental method in terms of reproducibility and performance.

In order to use samples as identical as possible, six samples of wire were taken from adjacent lengths of the same superconducting wire. 1/2 inch long pieces were then cut from each sample wire and mounted metallographically with an appropriate cross section for the image analysis. The sample wires were weighed and mounted on the resistivity apparatus for electrical measurement. After the electrical measurement was completed, the same wires were used for the chemical etching measurement.

2.2 Chemical Etching and Weighing

2.2.1 Basic Experimental Technique

From the chemical etching technique, the Cu/SC ratio is defined in terms of volumes of the respective components and measured by weighing a length of the composite wire before and after etching the copper matrix out of the wire. The Cu/SC ratio is calculated from the formula below:

$$Cu/SC = \frac{(\text{weight of copper} / \text{density of copper})}{(\text{weight of filaments} / \text{density of filaments})} \quad (1)$$

Or, this can be rewritten by four quantities as follows.

$$Cu/SC = \left[\frac{A\,\delta_{cu}}{M_w - M_{sc}} - 1 \right]^{-1} \quad (2)$$

where

M_w = the wire mass per unit length

M_{sc} = the mass of superconductor filaments per unit length

A = cross section area of the wire

δ_{cu} = density of copper matrix

A Nb-Ti multifilamentary composite wire with the copper matrix partially etched off is shown in Figure 4. During the fabrication process, just before the final size, the composite wire is twisted to reduce the coupling between the filaments.

Figure 4. A twisted filamentary Nb-Ti composite wire with the copper matrix partially etched away to reveal the Nb-Ti filaments. 50/50 mixture of HNO_3/H_2O etchant was used.

2.2.2 Test Procedure

Two sets of four 112 cm long SSC samples were tested after they had been used for electrical resistivity measurement. The test procedure is as follows.

A clean wide and deep dish was prepared and labeled with numbers for samples to be tested. The sample wires were cleaned carefully with methanol. The samples were coiled and the ends tucked securely over two or three times so that wires could not loosen during the etching. Tweezers were used to handle the samples for the rest of the testing procedure. Each wire sample was weighed with a microbalance to the nearest 0.1mg. Several measurements for each sample were made to get the weight average. The samples were then put in the dish with space enough between the samples to prevent them from tangling together at the beginning of etching. A 50/50 mixture of HNO_3/H_2O etchant solution was poured into the dish to cover the wire samples under the fume hood. It was necessary to keep the samples in the etchant at least an hour until all the copper is completely removed from the wires. Each wire sample was checked to see if all of the copper is etched off. This was done by checking the stiffness of the wires. If the etching action stopped but copper still remained, then the solution was replaced with fresh etchant. The filaments in the dish were gently rinsed with fresh water to avoid washing away any filaments. The filaments were put in the oven at 60°C-80°C for approximately 15 minutes. After the filaments were completely dry, weight measurement for each of the filaments samples were made to the nearest 0.1mg. Using the filament and copper weights, the Cu/SC ratio was calculated from Equation (1). For Nb-46.5wt.%Ti superconducting composites, the density of copper = 8.96 g/cm^3, and the density of Nb46.5Ti = 6.02 g/cm^3. Thus Equation (1) yields,

$$Cu/SC = 0.672 \frac{W_{cu}}{W_{sc}} \qquad (3)$$

The weight measurements for each sample were made three times to get an average value. Each sample was weighed in three stages: before mounting the samples on the probe for electrical measurement, after demounting the samples from the probe, and finally after etching off the copper matrix. With the chemical etching technique the calculation of the Cu/SC ratio was performed and corrected to take into account the effect of the Nb barrier which consists of an area equal to 4% of the Nb-Ti monofilament area. This is because the barrier is insoluble in nitric acid, so it remains with Nb-Ti filament after the copper is etched away.

2.3 Electrical Resistivity

2.3.1 Basic Experimental Technique

The basic theory behind the electrical resistivity measurement models the composite as a set of parallel resistors and was first worked out by Sampson and Garber [Ref 3]. If one knows the electrical resistivity of the components of the composite, from the electrical resistance per unit length of composite wire measured at both room temperature and liquid helium temperature it is possible to derive the cross section area of each component of the composite superconducting wire.

Since the composite wires consist of superconducting filaments with a parallel conducting path of copper, the resistance of the wire, R_w, can be expressed in terms of R_{cu} and R_{sc}, resistance of copper matrix and superconductor, respectively.

$$\frac{1}{R_w} = \frac{1}{R_{cu}} + \frac{1}{R_{sc}} \tag{4}$$

where

$$R_{cu} = \rho_{cu}\frac{L}{A_{cu}}, \qquad R_{sc} = \rho_{sc}\frac{L}{A_{sc}}$$

$$A_{cu} = A_w \cdot f_{cu}, \qquad A_{sc} = A_w \cdot f_{sc}$$

$$\rho_{cu,sc} : \text{resistivity}, \qquad f_{cu,sc} : \text{volume fraction}$$

Hence

$$\frac{1}{R_w} = \frac{A_w \cdot f_{cu}}{\rho_{cu} \cdot L} + \frac{A_w \cdot f_{sc}}{\rho_{sc} \cdot L} \tag{5}$$

The volume fractions can be written as a function of copper to superconductor ratio (Cu/SC).

$$f_{sc} = \frac{1}{Cu/SC + 1}, \qquad f_{cu} = \frac{Cu/SC}{Cu/SC + 1}$$

Substituting these into Equation (5) gives,

$$\frac{1}{R_w} = \frac{A}{\rho_{cu} \cdot L} \cdot \frac{Cu/SC}{Cu/SC + 1} + \frac{A}{\rho_{sc} \cdot L} \cdot \frac{1}{Cu/SC + 1}$$

that is [Ref 3],

$$Cu/SC = \frac{1 - [R_w A / \rho_{sc}]}{[R_w A / \rho_{cu}] - 1} \tag{6}$$

where R_w = resistance per unit length at the reference temperature of 295K

A = cross section area of the sample wire

It is normally true that the room temperature variations can produce significant variations in the measured resistance. In order to minimize the room temperature effect, the resistance at the reference temperature of 295K can be calculated from the expression below by designating the measured resistance at ambient temperature as R_a and the ambient temperature as T (°C).

$$R_{295} = R_a / [1+0.0039\,(T-22)]$$

For the purpose of this temperature correction, the effect of the Nb-Ti filaments on the resistivity is neglected.

2.3.2 Residual Resistivity Ratio

Measurement of the resistance per unit length for each sample wire was made at both ambient temperature (295K) and just above the transition temperature of Nb-Ti superconductor (10K). These two quantities are designated as R_{295} and R_{10}, and the ratio of R_{295}/R_{10} is then defined to be the residual resistivity ratio (RRR). This ratio serves as a general indicator of the resistance resulting from impurities and defects in the copper. Since, in large part, the resistance is determined by the copper matrix, one needs to know how pure the copper is in order to determine the ρ_{cu} used in Equation (6).

Figure 5 shows the relationship between electrical resistivity and temperature for the copper, with varying RRR value [Ref 8]. All metals have a tendency to decrease in electrical resistivity as temperature is lowered. However, even though the temperature goes down to below 10K for copper, the resistivity will not decrease any more due to any impurities and defects in the copper. Ideally the ratio, RRR, approaches infinity as the resistivity at low temperature approaches zero indicating the material is nearly pure.

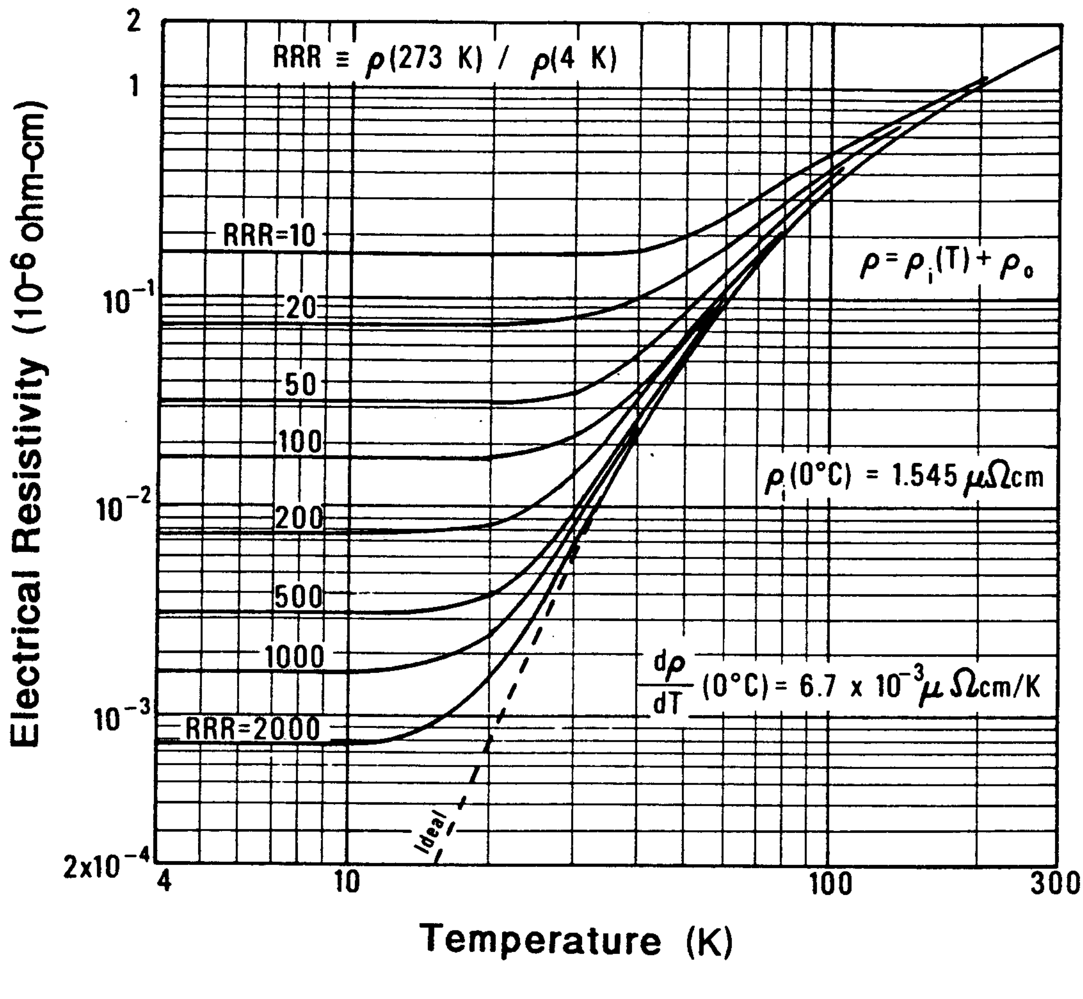


Figure 5. Electrical resistivity of the copper plotted against temperature.

2.3.3 Determination of Cu/SC Ratio

The copper to superconductor volume ratio can be calculated from measured resistance at both ambient temperature and low temperature made in a helium dewar.

$$Cu/SC = \frac{1 - [R_a\, g(T)\, A / \rho_{sc}]}{[R_a\, g(T)\, A / \rho_{cu}] - 1} \qquad (7)$$

where

R_a = measured resistance per unit length at ambient temperature (ohm)

$g(T)$ = temperature correction factor = $1 / [1+0.0039\,(T-22)]$

A = cross section area of the wire (cm^2)

ρ_{sc} = resistivity of Nb-Ti at 295K

= 60×10^{-6} ohm-cm

ρ_{cu} = resistivity of copper at 295K (ohm-cm)

= $\rho_i\, [RRR / (RRR - 1)]$

where ρ_i = resistivity of pure copper at 295K

= 1.75×10^{-6} ohm-cm (cold worked)

= 1.71×10^{-6} ohm-cm (annealed)

RRR = $R_a\, g(T) / R_{10}$

R_{10} = measured resistance per unit length at 10K

2.3.4 Apparatus Description

The measurement of the electrical resistance of the sample wires is based on the measurement of the V-I curve. While an increasing DC current is passed through the superconducting wire sample, the voltage across the sample is simultaneously measured between the voltage taps soldered to the sample. A four wire method is used to determine the resistance and the wire samples are mounted on a former connected at the end of a probe which has two types of leads; one for carrying the required current from room temperature into a liquid helium bath, one for measuring the voltage across the measured length of the test sample. A schematic diagram of the electrical measurement system used in this experiment is shown in Figure 6.

The sample current in the range from 0 to 5A is provided by a well-filtered DC power supply, and is measured by a very accurate shunt. The voltage across the current shunt is measured with a Keithley 177 Microvoltmeter. The scaled output voltage is then sent to the Macintosh IIci computer data acquisition system.

The voltage drop between the two voltage taps on the wire sample is detected on a Keithley 155 Microvolt Null Detector with measurement resolution of 0.5 μV. This voltage difference is then transferred to the computer to obtain the voltage signal. For these wire samples voltage ranges from 0 to 0.03V at room temperature, and from 0 to 0.006V at low temperature were measured in this way.

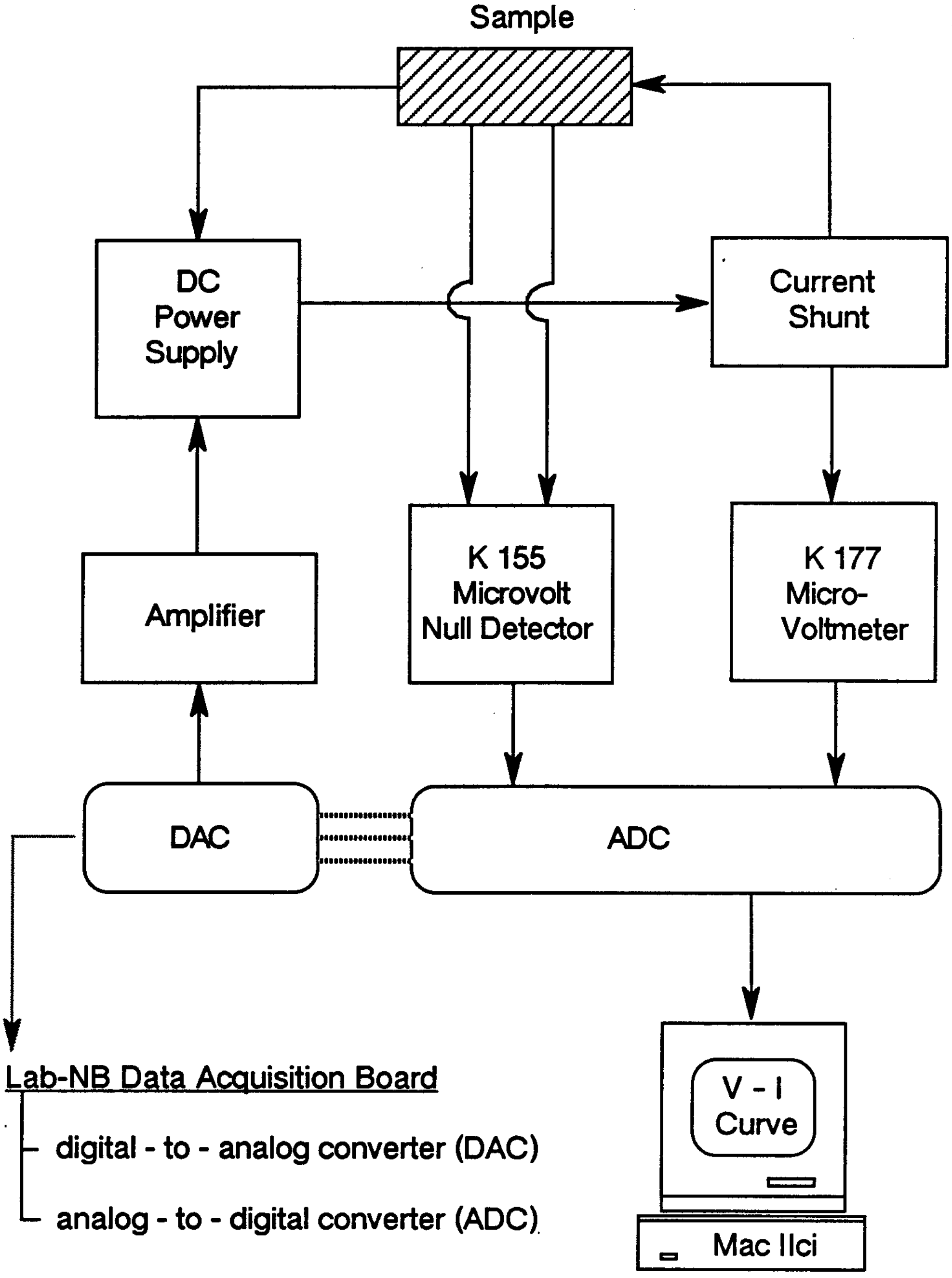


Figure 6. A block diagram of the electrical measurement system used in this experiment.

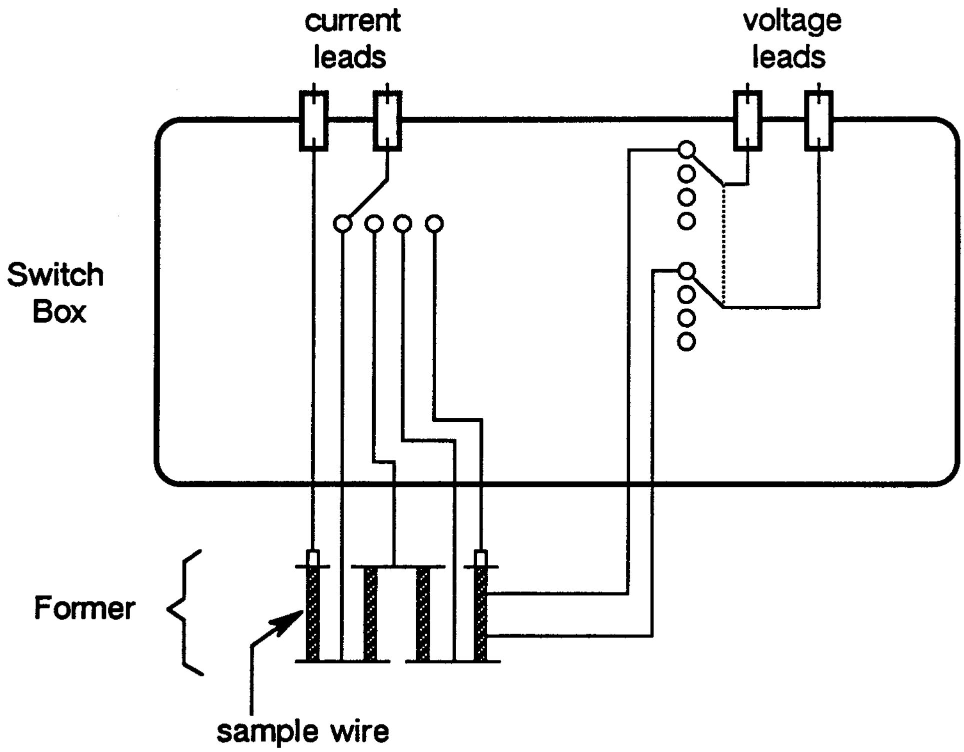
A computer program written in Lab VIEW 2.0 software is used with a Macintosh data acquisition system to control the experiment, collect the raw data, and then calculate the electrical resistance.

The four samples are cut to 112 cm long and wound on a spirally grooved former in order to securely locate the samples in place (see Figure 7). A cylindrical G-10 former is used to electrically isolate the four wire samples from one another. The ends of the samples are secured with nuts to the copper terminals of the current leads.

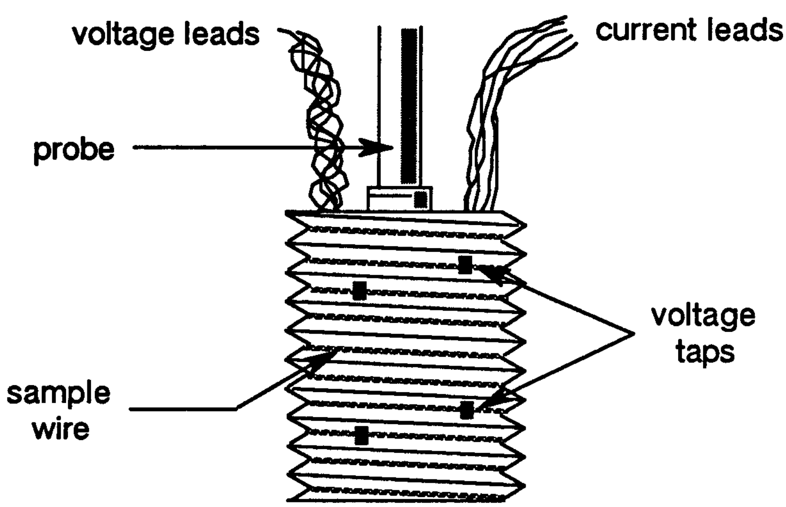
Each of the four sets of voltage taps is soldered to the corresponding sample wire at a separation distance of 65.4 cm between the taps. All the taps are far enough from the current joint near the ends of the samples to prevent voltages due to transferring current. It is also desired that these taps should be fixed to the former so that the test length is constant through a series of measurements. The voltage taps consist of eight twisted leads, and they are co -wound with the samples to reduce inductive voltage loops.

2.3.5 Test Procedure

The electrical resistance measurements at room temperature are made within a range of currents between the minimum requirements of sensitivity and negligible ohmic heating. Typical current values for this type of sample were found between 0 and 0.8A. Voltage readings were taken with increasing and decreasing current and averaged to get a slope which equals the resistance.



(a)



(b)

Figure 7. (a) Circuit diagram of switch box and former.

(b) Sample mounting arrangement for electrical method.

This eliminates any thermocouple or amplifier voltage offset and provides a very accurate measure of the resistance.

Low temperature measurements are made in a nitrogen shielded helium dewar. The probe is withdrawn from the helium bath so that the samples are located just above the liquid helium level while voltage is being measured. As the sample wire warms, the voltmeter reading goes suddenly from zero to a big value indicating normal state resistance. Since this phenomenon, however, is independent of temperature from the transition temperature (10K) to 15K, it is practically possible that the voltage remains constant long enough to be read in this stage.

2.4 Metallographic Image Analysis

2.4.1 System Description

The useful geometric parameters for the composite superconducting wires can be obtained directly by examining the cross section of the wires using metallographic image analysis with the optical or electron microscope. Digital image processing and analysis using Image Analyst™ software with Macintosh IIci consists of extracting useful information about objects by mathematically manipulating the gray scale value of pixels in a buffer and using a variety of statistical analyses. An image can be acquired either from a video camera attached to an optical microscope, or from a disk based image file. The magnifications to be used are determined based on an optimization for either the interfilamentary region or the whole wire area. The photographs obtained are then digitized through a video digitizer and transferred to the image analysis software. This procedure needs to be repeated for several photographs taken from different locations in the cross section of the sample wire and final analysis is done by considering all these results. A basic flow chart of analyzing images using Image Analyst is illustrated in Figure 8.

Once an image is acquired from a video camera, a new image window is created with a size of 600 pixels wide by 512 pixels high. Each pixel is assigned a gray value between 0 and 255 (0 for black and 255 for white) determined by the digitized image. The matrix of image pixels with their gray scale values is sent to the image analysis software to perform basic image processing including sharpening, increased contrast, edge enhancement, smoothing, and others. A threshold gray scale value is then selected to differentiate between filament and matrix pixels. All pixels with gray level below the threshold are set to be black to represent the filaments, and above the threshold are set to be white to denote the copper matrix. The area ratio of the components is then measured by counting the total number of black and white pixels. The connectivity tool interprets the areas of continuous black pixel as discrete objects from which other geometrical parameters such as average area, shape, and spacing of the filaments can be measured.

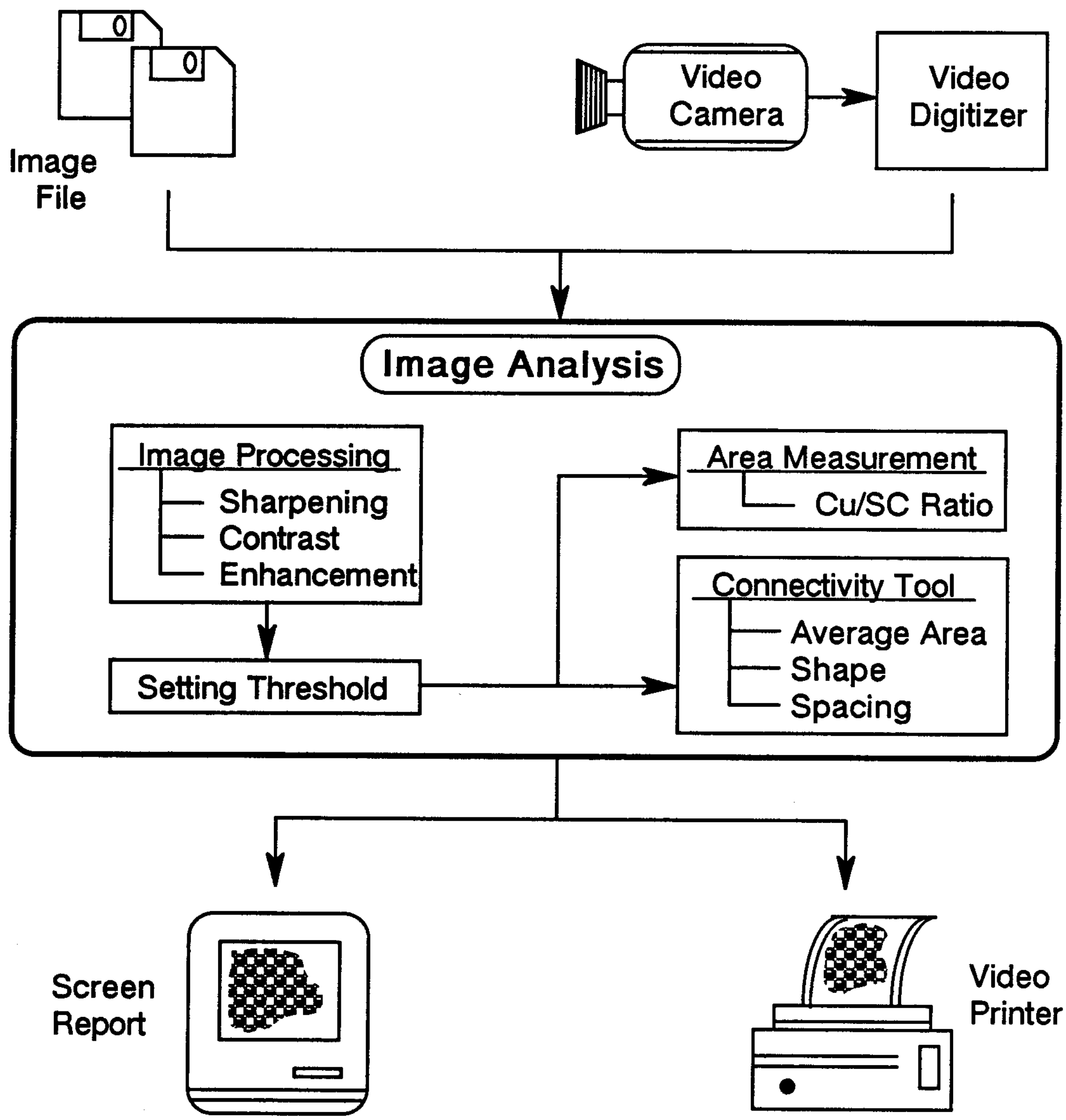


Figure 8. Overview of image processing with image analysis.

2.4.2 Sample Preparation

Several half inch long pieces of wire were removed from each sample wire and metallographically mounted in an phenolic bakelite base with transverse cross sections for image analysis. The samples were ground using progressively finer grit sand paper. Polishing wheels were then used to finish polish the surface of the samples. The abrasive used was Al_2O_3 at 5, 0.3, and 0.05 µm in diameter. Etching was done using a mixture of $10HF/5HNO_3/85H_2O$ for 10-20 seconds, which provides a strong contrast between the filament and the copper matrix. This etch was found to work well for optical microscopy as well as deep etching the Nb-Ti filament to give a surface profile easily observed by the scanning electron microscope (SEM). For the purpose of access to the SEM, a layer of conductive bakelite must be added on the sample mount.

2.4.3 Area Measurement

The area measurement of the filaments and the matrix in the cross section of the sample wires were performed with stored images during the image analysis. Since the gray scale values for the filaments and the matrix are remarkably different (see Figure 9), a gray level was selected by setting the appropriate threshold which should be at the boundary between filaments and matrix.

Figure 10 shows a plot of the frequency versus gray value made from the stored image using the histogram tool. There are two distinct peaks, one for the filaments and the other for the matrix.

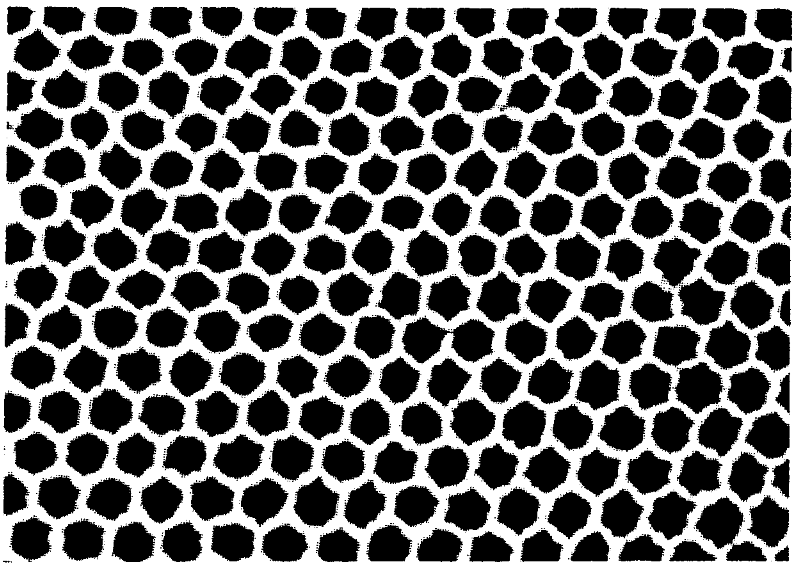


Figure 9. A binary image made from the interfilamentary region.

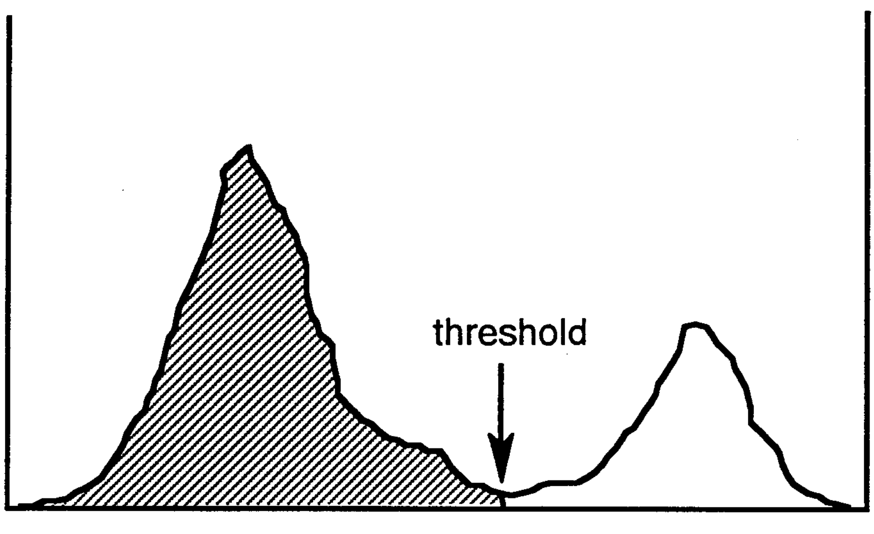


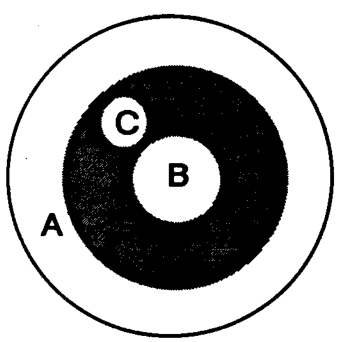
Figure 10. A histogram of pixel frequency vs. gray value made from a stored image based on the binary system. A threshold gray value is chosen at the minimum between the two peaks to define the boundary between the filaments (shaded) and the matrix.

A binary image was created in which pixels with gray values below the threshold were set to be black to define the filaments and all remaining pixels was set to be white to represent the matrix. The area of the filaments and the matrix was then measured as a number of pixels occupying the respective components, from which the Cu/SC ratio was simply calculated for both local and overall areas.

The overall Cu/SC ratio measurement was performed using the formula below with the cross section geometry of the sample wire (see Figure 11).

$$Cu/SC = \frac{A + B + C \cdot f_{cu}}{C(1 - f_{cu})} \qquad (8)$$

where f_{cu} is the area fraction of interfilamentary copper in region C. Figure 12 represents the microphotographs of overall area and interfilamentary region in cross section of the sample wire.



A, B – Bulk Copper

C – Interfilamentary Region

Figure 11. Schematic geometry of cross section of the sample wire.

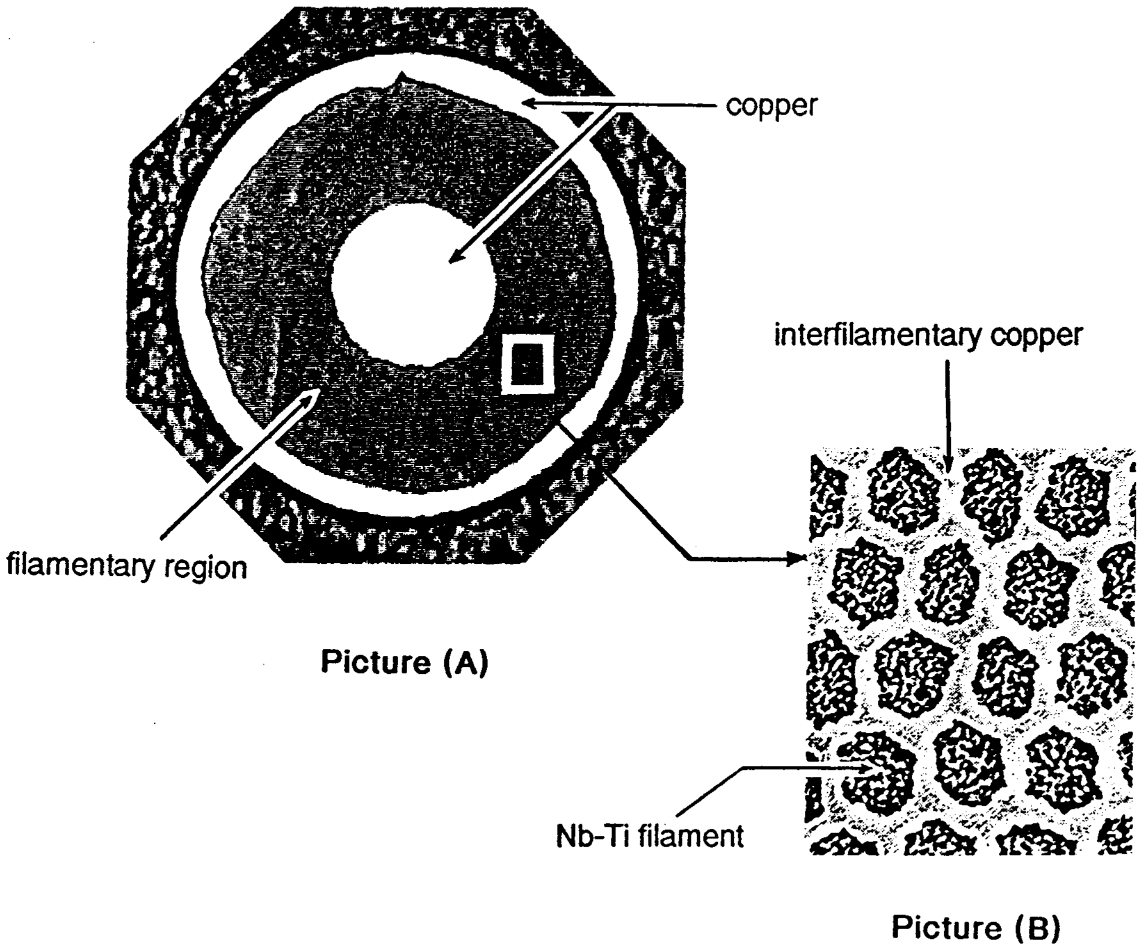


Figure 12. Microphotographs of cross section of the sample wire. Pictures were taken from optical microscope (A), and SEM (B).

2.4.4 Advantages

The geometric parameters in cross section of a superconducting wire have been considered to be important for both electrical and mechanical stabilities of final products. In this respect, image analysis is a very powerful technique because it is capable of providing much more information on the wire geometry, as well as being fairly easy to perform through a non-destructive sampling procedure. That is interfilamentary Cu/SC ratio, filament diameter (D), and spacing between the filaments (S) can be determined using image analysis. Besides, this technique can be automated for quick measurements, and shows fair repeatability with a small amount of sample consumption and easy preparation for standard metallographic sample mounting.

Measurements of filament diameter and spacing for all samples were made with the optical microscope during image analysis. Suitably sized sample wires were mounted and observed on the optical microscope using the video camera. By using a stage micrometer the relation between pixels and millimeters was found. Ten measurements of filament diameter and spacing were taken and averaged to get the final results. Spacing between the filaments was defined as the nearest distance between two regularly shaped filaments.

CHAPTER 3 EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Chemical Etching

Two sets of four 112 cm long SSC sample wires were tested by chemical etching after they had been used for electrical resistivity measurement. Measurement data are shown in Table 1. Each sample was weighed in three stages: before mounting, after demounting, and finally after etching off the copper. Each measurement was made three times to get an average value.

Table 1. Weight measurement data and the calculated Cu/SC ratio.

Sample No.	Before Mounting (grams)	After Demounting (grams)	After Etching (grams)	Cu/SC Ratio
1	4.3628	4.3647	1.5578	1.302
2	4.4469	4.4502	1.5903	1.299
3	4.4377	4.4394	1.5861	1.300
4	4.4679	4.4706	1.5976	1.299
5	4.3846	4.3877	1.5639	1.304
6	4.3513	4.3540	1.5537	1.302
7	4.4232	4.4248	1.5769	1.305
8	4.3955	4.3969	1.5678	1.304

The differences in weight measured in between the first and the second stages have been found to be less than 0.07%, which can be negligible. This allows the results from either of these two stages to be used to calculate the Cu/SC ratio in Equation (3).

3.2 Electrical Resistivity

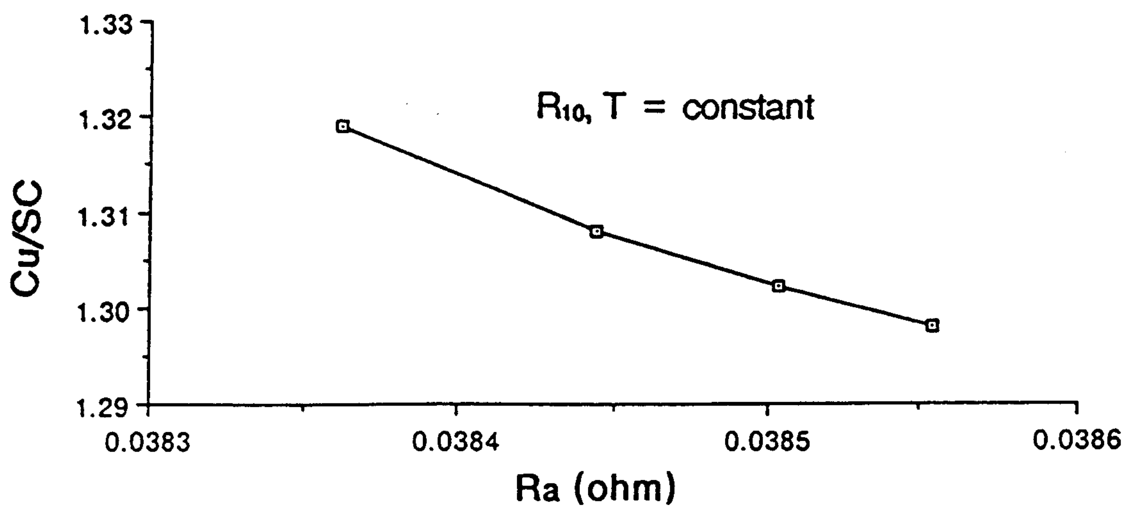
As it appears in Equation (7), the Cu/SC ratio can be expressed as a function of ambient temperature and measured resistance at both room and helium temperature in electrical resistivity method. The calculated average value of RRR for the sample wires was found to be 46.3. Table 2 shows the data obtained from this technique.

Table 2. Resistance measurement results for the sample wires.

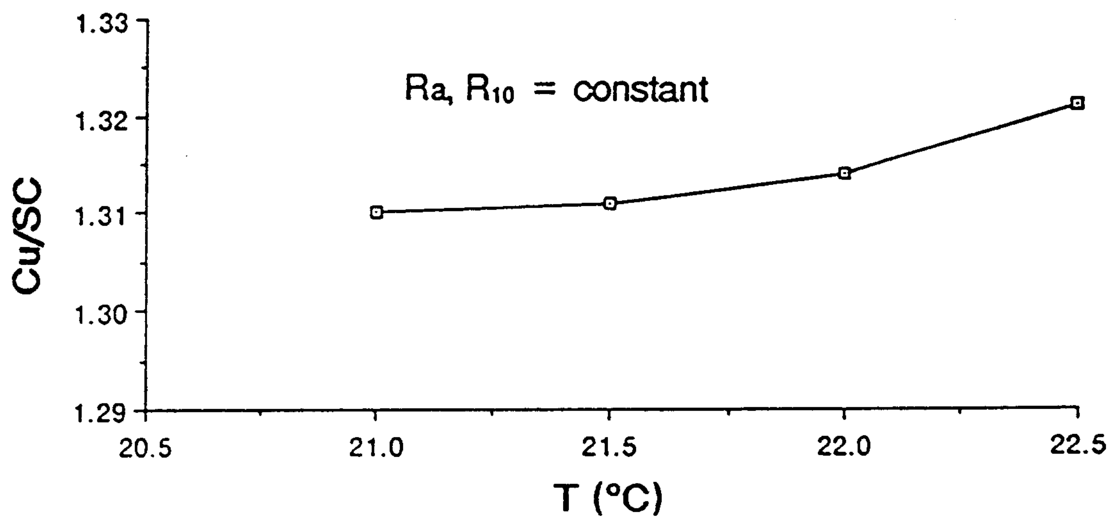
Sample No.	Diameter (mm)	T_a (°C)	Resistance (ohm/cm) R_a	(x10^{-4}) R_{10}	Cu/SC Ratio
1	0.81	20.0	5.995	1.283	1.28
2	0.81	21.0	5.823	1.233	1.38
3	0.81	20.5	6.016	1.317	1.27
4	0.81	21.0	6.025	1.320	1.28
5	0.81	20.5	6.101	1.283	1.23
6	0.81	21.0	6.076	1.233	1.25
7	0.81	21.0	6.077	1.317	1.25
8	0.81	21.5	6.077	1.320	1.26

3.2.1 Sensitivity to the Measurement Errors

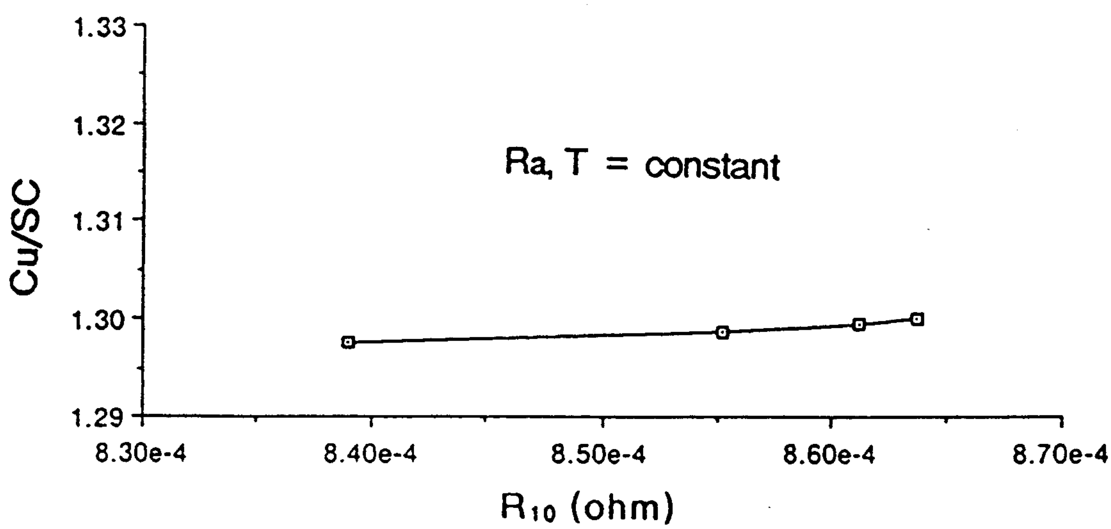
Figure 13 shows the sensitivity of the electrically determined Cu/SC ratio to measurement errors. This analysis was done by investigating the range of the Cu/SC ratio varying one of the variables with the other two variables fixed. The values for each variable in x-axis were selected from a set of possible measured data in this experiment. By observing these plots, it is obvious that the electrical resistance at ambient temperature represents the most sensitivity to the Cu/SC ratio.



(a)



(b)



(c)

Figure 13. Sensitivity of electrically determined Cu/SC ratio to the measurement errors: (a) resistance at ambient temperature, (b) ambient temperature, and (c) resistance at low temperature.

3.2.2 Uncertainty

Since the expression of the Cu/SC ratio is a function of three independent variables for electrical method, the total errors in the Cu/SC ratio are governed by these three factors: the ambient temperature (T), resistance at both ambient (R_a) and low temperature (R_{10}). Using the formulas for the error estimation[Ref 12], the total uncertainty in the result can be estimated by combining the standard deviation of each independent variable.

The error analysis results represent that the total uncertainty in the Cu/SC ratio is in the range between 5% and 12%. The resistance at ambient temperature is the most powerful factor affecting the Cu/SC ratio measurement.

3.3 Image Analysis

The local and overall Cu/SC ratio calculated from image analysis process are shown with another geometric parameters of these sample wires in Table 3.

It has been seen that the result of the overall Cu/SC ratio shows fair reproducibility even though the local Cu/SC ratio result shows the more scatter. The results from spacing and diameter measurements for all the samples agree within 0.1% and 0.2%, respectively.

Table 3. Geometric parameters data obtained from image analysis.

Sample No.	Overall Cu/SC	Local Cu/SC	D (μm)	S (μm)
1	1.27	0.77	6.3	1.1
2	1.28	0.62	6.0	1.3
3	1.30	0.71	6.4	1.0
4	1.28	0.74	6.2	1.1
5	1.32	0.58	6.4	1.0
6	1.28	0.61	6.2	1.2

3.4 Comparison in Techniques.

Experimental results of the Cu/SC ratio measurements obtained from the three experiments are shown in Table 4. The average results from all these techniques agree within 2%, however there is a large difference in the standard deviation between the techniques. The chemical etch & weigh technique shows the smallest variation, with a S.D. of less than 0.2%. Image analysis shows the next smallest scatter, with a S.D. of about 1.5%, and electrical method shows a scatter of nearly 4%.

The results from the three techniques can be summarized as follows:

(1) The etching & weighing technique is easy to perform, with a low level of technical expertise required, small equipment costs, and gives by far the best reproducibility. The only drawback is it is a destructive test.

(2) The electrical resistivity technique, by contrast, is difficult to perform, requires liquid cryogen with a high level of technical expertise, and produces the most variability in the results. Sensitivity analysis indicates a large sensitivity to the room temperature resistance measurements, which is difficult to overcome.

(3) Image analysis is a surprisingly good technique and shows fair repeatability with small sample consumption and easy preparation. This technique can be automated for quick measurements.

Table 4. Comparison in the Cu/SC ratio measurements from the three techniques.

Sample No.	Cu/SC Ratio		
	Chemical	Electrical	Image
1	1.302	1.28	1.27
2	1.299	1.38	1.28
3	1.300	1.27	1.30
4	1.299	1.28	1.28
5	1.304	1.23	1.32
6	1.302	1.25	1.28
7	1.305	1.25	–
8	1.304	1.26	–
Average	1.302	1.28	1.29
S. D.	0.002	0.05	0.02

CHAPTER 4 CONCLUSIONS

Chemical etching and weighing was found to be the most reproducible and simple technique for determining the Cu/SC ratio, whereas the electrical resistivity method showed the most variation. From investigating the dependence of the Cu/SC ratio on the variables in electrical resistivity techniques, it has been seen that the ambient temperature resistance produced the largest variation in the Cu/SC ratio. The metallographic image analysis method is fairly consistent with the traditional etch and weigh technique, and is capable of providing much more information on the wire parameters than either of the other techniques. Based on this study, use of the electrical resistivity measurement for Cu/SC ratio determination should not be encouraged.

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