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THERMAL CONTRACTION OF FIBERGLASS-EPOXY SAMPLE

HOLDERS USED FOR Nb₃Sn CRITICAL-CURRENT MEASUREMENTS*

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ABSTRACT

It is typical for Nb₃Sn-Cu superconductor specimens to be wound into coils on tubular specimen holders for critical-current measurements. If the thermal contraction of the holder is different than that of the specimen, axial strain may be applied to the specimen upon cooling from room to liquid-helium temperature. This strain can affect the measured critical current. The thermal contraction was measured for three different Nb₃Sn-Cu superconductors. Also, the thermal contraction was measured for several different specimen holders, all of which were made from fiberglass-epoxy composites. The specimen holder measurements were made using an electrical-resistance strain-gage technique, and they were confirmed by direct mechanical measurements. The tubes varied in diameter, wall thickness, and fabrication technique. Some of the tubes were made directly from tube stock, and others were machined from plate stock. The results of these measurements show that the thermal contraction of tube stock is strongly dependent on the ratio of its wall thickness to its radius, while the contraction of tubes machined from plate stock is relatively independent of these dimensions. Critical-current measurements of Nb₃Sn-Cu specimens mounted on these various holders show that the presence of differential thermal contraction between the specimen and its holder can significantly affect the measured critical current.

INTRODUCTION

Critical-current (I_c) measurements of Nb₃Sn-Cu superconductors can be affected by the relative thermal contraction of the material on which the test specimen is mounted. This measurement variable became apparent in the recent VAMAS (Versailles Project on Advanced Materials and Standards) interlaboratory comparative measurements (round robin) of the critical current of Nb₃Sn.¹ Differential thermal contraction between the specimen and its holder in cooling from room to liquid-helium temperature can cause either a tensile or compressive strain of the specimen, either of which can affect the measured I_c .^{2,3} For coil specimens that are mounted on the surface of cylindrical holders, the strain is predominantly along the axis of the specimen. The amount of strain depends on the magnitude of the

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differential contraction, the relative strengths of the specimen and holder, and the mechanical coupling between the specimen and its holder.

The literature contains considerable data on the compressive pre-strain of Nb_3Sn filaments caused by differential thermal contraction between the filaments and the matrix material. However, very little data on the overall thermal contraction of Nb_3Sn -Cu wires are presently available. The thermal contraction of a Nb_3Sn -Cu cable is given in Ref. 4; however, this conductor has a tungsten core that reduces its thermal contraction. Consequently, the thermal contraction of three different Nb_3Sn wires was measured as a part of this study.

Fiberglass-epoxy composites are commonly used sample holder materials. These materials are anisotropic in three mutually perpendicular directions. The three directions are associated with characteristics of the fiberglass fabric, and they are designated as the warp, fill, and normal directions. The fabric orientation for plate stock is shown in Fig. 1. The normal direction is perpendicular to the fabric planes, while the warp and fill directions are parallel to the fabric planes. The density of the fabric is not the same in both directions of the weave. The warp and fill directions are determined by the fabric's thread count. The number of threads per unit length of fabric is lower in the warp direction than in the fill direction. This structural anisotropy causes a three-dimensional variation in thermal contraction. The contraction in the fill direction is slightly greater than in the warp direction, but the contraction in the normal direction is considerably larger.^{4,5}

Figure 1 also shows the typical fabric orientation for a rolled tube. The radial thermal contraction of the tube results from a competition between the larger contraction in the normal direction and the smaller contraction in the warp direction. Consequently, it depends on the ratio of the tube's wall thickness to its outside radius (wall-to-radius ratio). For thin-walled tubes the radial thermal contraction approaches that of a plate in the warp direction, and for thick walled tubes it approaches that of a plate in the normal direction.

A holder whose thermal contraction is relatively independent of its wall-to-radius ratio can be made by machining a cylindrical tube from thick fiberglass-epoxy plate stock with the axis of the tube perpendicular to the surface of the plate (plate tube). For this orientation (Fig. 1) the radial contraction is based on the contraction in the warp and fill directions,

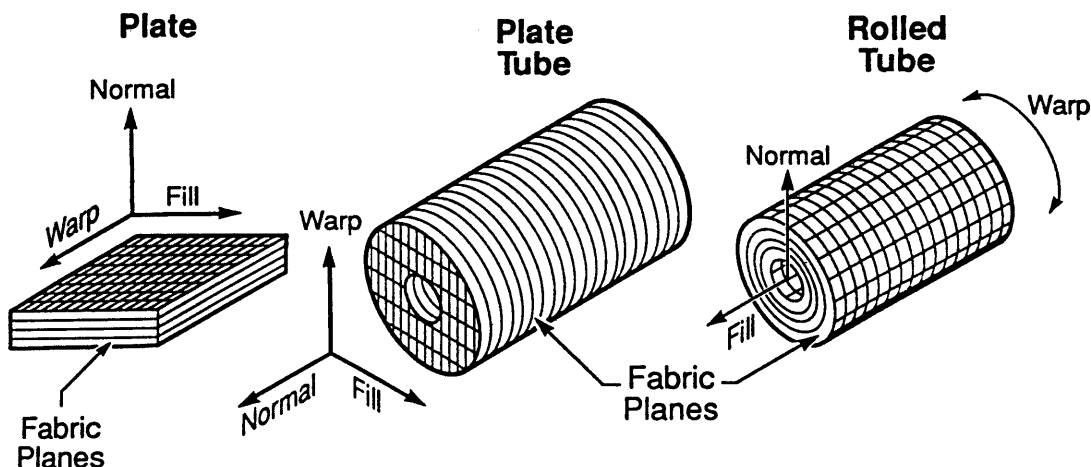


Fig. 1. Geometries of fiberglass-epoxy composites.

which are both similar to that of a Nb₃Sn-Cu wire. In addition to thermal contraction measurements of both rolled tubes and plate tubes, I_c measurements were made using both types of specimen holder to confirm the relationship between thermal contraction and the I_c measurement.

EXPERIMENTAL DETAILS

Fiberglass-Epoxy Composites

Two different types of fiberglass-epoxy composite were used in this study, NEMA (National Electrical Manufacturers' Association) G-10 and G-11. All of the rolled tubes were G-10 and the plate tubes were G-11. This was not a matter of choice but, rather, one of material availability. The thermal contraction of the G-11 is slightly less than that of G-10. Neither of these materials was cryogenic-radiation (CR) grade, which is designated as G-10CR or G-11CR. The manufacturing specifications for the CR grade materials are more stringent and their performance at cryogenic temperatures is more predictable.⁵

Thermal Contraction

All of the thermal contraction measurements were conducted between room temperature and liquid-nitrogen (LN₂) temperature. We assume that, if a material's contraction is well matched to a Nb₃Sn-Cu wire from room to LN₂ temperature, then the additional differential thermal contraction that occurs between LN₂ and liquid-helium temperature will be insignificant.

The Nb₃Sn-Cu thermal contraction measurements were made using quartz reference tubes and reacted Nb₃Sn-Cu specimens that measure approximately 35 cm in length. The specimen is placed in a quartz tube and attached to one of the tube's ends. At room temperature, the quartz tubes are approximately 1 mm longer than the specimens. This differential length is measured with a micrometer, the tube and specimen are cooled to LN₂ temperature, and the differential length is remeasured. The thermal contraction of the specimen is deduced from the measured change in the differential length and the known contraction of the quartz tube. The uncertainty of these measurements is estimated to be ±5%.

Two different methods were used to measure the thermal contraction of the fiberglass tubes. The first was a mechanical method, where the tube was submerged in liquid nitrogen, allowed to reach thermal equilibrium, removed from the nitrogen, and the diameter was then quickly measured with a precision micrometer. To address the thermally transient nature of these measurements, the tube's diameter was measured as a function of time while it was warming toward room temperature. These measurements allowed an extrapolation of the data to liquid-nitrogen temperature. In the case of the plate tubes, two orthogonal measurements were made at each time, one in the warp direction and one in the fill direction. The uncertainty of the mechanical measurements of thermal contraction was estimated to be ±10% of the measured value (for example, 0.20% ±0.02%).

The second type of thermal contraction measurement used electrical-resistance strain gages.⁶ For this measurement, two well matched 350 Ω strain gages were used in a half-bridge configuration. One of the gages was bonded to the test specimen and the other was bonded to a reference material, a quartz tube, whose thermal contraction was known. All of the strain-gage measurements reported here were taken with the strain gages mounted on the circumference of the test specimen since this represents the contraction which is relevant for the Nb₃Sn-Cu coil. Both the test specimen

and reference material were cooled to liquid nitrogen temperature and the resulting output from the strain bridge was measured.

Two factors will cause a change in the resistance of the strain gages when cooled from room to LN₂ temperature. First, the resistivity of the strain gages' grid alloy will change with temperature. Since this change in resistance will be nearly equal for matched strain gages, there will be little effect on the output of the strain bridge. The second source of change in resistance is caused by thermally induced strain. This strain results from differential thermal contraction between the strain gage and the test specimen. If the thermal contraction of the test specimen material is different than that of the reference material, the strain bridge will be unbalanced and its output will be indicative of the difference in thermal contraction between these two materials. Since the thermal contraction of the reference material is known, the thermal contraction of the test material can be deduced. The uncertainty of the strain-gage measurements of thermal contraction was estimated to be $\pm 5\%$ of the measured value (for example, $0.20\% \pm 0.01\%$).

Critical Current

For the I_c measurements, a Nb₃Sn-Cu wire with a diameter of 0.68 mm was used. This conductor was made by an internal-tin diffusion process, and it has 37 sub-bundles of 150 Nb filaments. A single Ta diffusion barrier separates the filament region from the outer Cu layer. The specimen was wound onto a stainless steel tube and then vacuum heat treated at 700°C for 48 h. The stainless steel tube has a helical groove machined into its surface to retain the specimen and define its geometry. Following heat treatment, the specimen was removed from the stainless steel reaction holder and transferred to the fiberglass measurement holder. The outside diameter of the fiberglass holder is 3.12 cm and its surface is not grooved. A thin continuous layer of filled epoxy adhesive was painted over the surface of the specimen and holder. The typical specimen length was approximately 80 cm. Three pairs of adjacent voltage taps were placed along the center of the specimen. Each pair had a separation of about 10 cm, and there was a 1 to 2 cm gap between adjacent pairs. An electric field criterion of 10 $\mu\text{V}/\text{m}$ was used for determining the critical current. The uncertainty of the I_c measurements may be as large as $\pm 5\%$ because of the strong systematic effects of the specimen holders.

RESULTS

Thermal Contraction Measurements

The results of the thermal contraction measurements are shown in Fig. 2 where the thermal contraction of the tubes' diameters ($\Delta d/d$) is plotted as a function of the tubes' wall-to-radius ratios. The thermal contraction of three different superconductors (the VAMAS I_c round robin conductors¹) was also measured. The dashed horizontal line labeled "Nb₃Sn-Cu" indicates the measured thermal contraction (0.28%) of the conductor whose I_c was measured in this study. The measured thermal contraction of the other two conductors is 0.28% and 0.26%. The two types of data symbols that are labeled "Mechanical" and "Strain Gage" show the thermal contraction of the G-10 rolled tube specimens as measured by the two different techniques. With the exception of the "Mechanical" data, all of the data shown in this figure were obtained using the strain-gage technique.

The data show a strong dependence of the thermal contraction on the wall-to-radius ratio. The approximate thermal contraction of G-10CR plate stock in cooling from room to liquid-nitrogen temperature is 0.224%, 0.264%,

and 0.687% in the warp, fill, and normal directions respectively, and it is 0.202%, 0.227%, and 0.585% for G-11CR.^{4,5} As expected from the structural geometry of the rolled tubes, their thermal contraction fall between that of the warp and normal directions of plate stock. The measured thermal contraction of the G-11 plate tubes is shown for three wall-to-radius ratios and in two structural directions, warp and fill. In both directions, the thermal contraction is relatively independent of the wall-to-radius ratio and it is comparable to that of plate stock. The error bars associated with the high wall-to-radius ratio data points represent the range of values that were measured on three different specimens with three repeat determinations for each specimen.

Additional measurements were made using strain gages that were mounted on the circumference of the plate tube halfway between the warp and fill axes. The thermal contraction at these positions was within the experimental uncertainty of the value measured in the warp direction. This indicates that the effective circumferential thermal contraction of the plate tube is closer to the value measured for the warp direction. Mechanical thermal contraction measurements were also made on the plate tubes and were within the experimental uncertainty of the strain-gage measurements. Additional thermal contraction measurements were made on tubes with different outer diameters to test the scaling of thermal contraction with the wall-to-radius ratio. All of the data of Fig. 2 were taken on tubes having outer diameters of about 3.18 cm and various inner diameters. However, two rolled tubes with outer diameters of 11.4 and

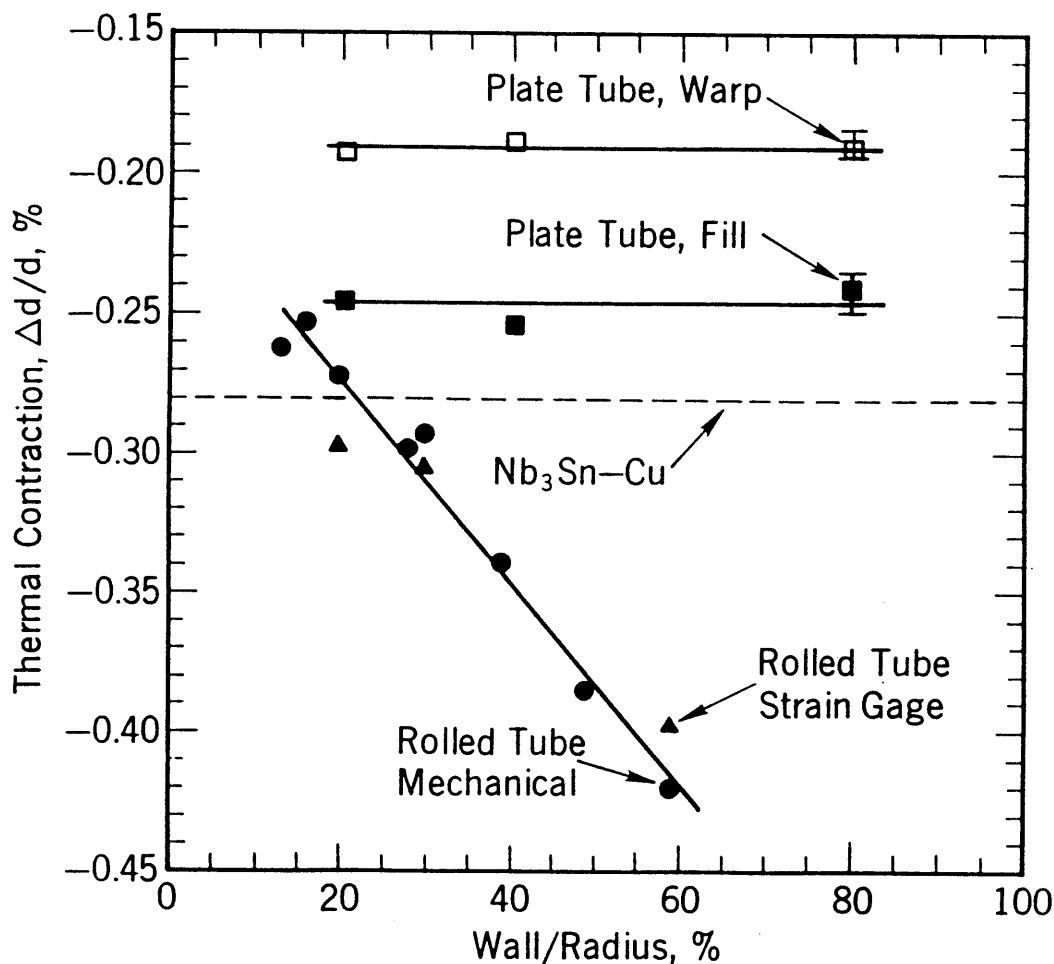


Fig. 2. Plot of room to liquid-nitrogen temperature thermal contraction of G-10 rolled tubes and G-11 plate tubes as a function of the tubes' wall-to-radius ratio.

12.4 cm, and wall-to-radius ratios of 14 and 8%, respectively, were also measured. The thermal contraction of these two tubes was within the experimental uncertainty of the rolled tube data of Fig. 2. Finally, an 11.4 cm outer diameter (94% wall-to-radius ratio) plate tube was measured and was within the experimental uncertainty of the plate tube data of Fig. 2.

Critical-Current Measurements

The results of the I_c measurements are shown in Fig. 3, where the I_c is plotted as a function of applied magnetic field for several different conductor specimens and specimen holders. There are three variables, in addition to the applied magnetic field, for these I_c measurements: the location where the specimen was reacted, "central" or "self"; the specimen holder's wall-to-radius ratio, "thick" or "thin"; and the type of tube used for the specimen holder, "rolled" or "plate". The self reacted samples were reacted at NIST, whereas the central reacted samples were reacted at another site and shipped to NIST for I_c measurements. The thick and thin designations do not indicate specific wall-to-radius ratios; instead, they indicate two general categories of tube geometry. All of the thin tubes have wall-to-radius ratios that are no greater than 13%

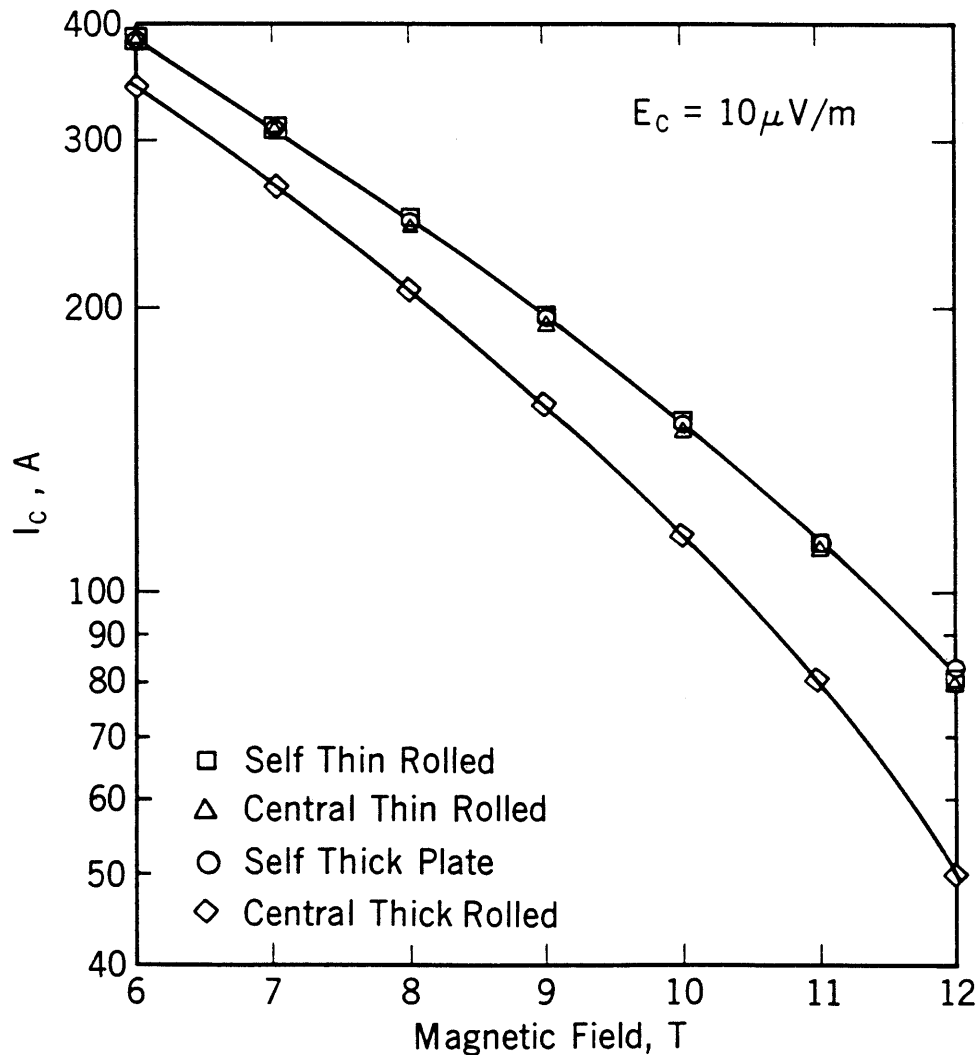


Fig. 3. A semilogarithmic plot of I_c at an electric field criterion of $10 \mu V/cm$ as a function of magnetic field.

(self 10% and central 13%) and the thick tubes have wall-to-radius ratios that are at least 60% (plate 80% and rolled 60%). The measured I_c 's are nearly the same, within experimental uncertainty, for all of the specimens except the one that was mounted on the thick, rolled tube. The measured I_c is significantly lower for this specimen at all magnetic fields. The difference is about 14% at 6 T and 40% at 12 T. The fact that the I_c degradation increases with increasing magnetic field is consistent with a strain effect. The magnitude of the change in I_c and the measured thermal contraction are in good agreement with the strain effect.³ At 12 T, a 33% reduction was calculated from strain-effect measurements¹ that were made at 14 T, and the measured I_c reduction was 40%.

DISCUSSION

A practical reality of round robin measurements is that the dimensions of the specimen holders vary between different laboratories. For consistent measurements, the specimen holders should be designed so that the I_c measurement is insensitive to this variable. The thermal contraction of a tubular specimen holder that is made from an anisotropic material can vary with its geometry. This presents the potential for variations in the strain state of the specimen and, thus, variations in the measured I_c . An apparent solution to this problem is to use an isotropic material for the specimen holders and to use a bonding technique that rigidly couples the specimen to its holder. This will ensure that the strain transmitted to the specimen, due to thermal contraction, is independent of the holder's geometry and, thus, equivalent from laboratory to laboratory. This approach addresses the issue of measurement consistency, but it does not address accuracy.

The I_c measurement should, arguably, be made with a minimum of externally applied strain on the superconductor. This requires a strong bond between the specimen and holder to avoid specimen strain under the influence of the Lorentz force, and it requires that the thermal contraction of the sample holder be well matched to that of the superconductor. Also, the holder should, ideally, be made from an electrically insulating material to prevent current sharing with the test specimen. Unfortunately, an isotropic and insulating material with a thermal contraction similar to that of Nb₃Sn-Cu is not readily available. Fiberglass-epoxy plate tubes are a practical alternative to the ideal isotropic specimen holder. The thermal contraction of a plate tube is slightly anisotropic; however, it is relatively independent of the tube's dimensions. Furthermore, the thermal contraction in the radial direction (the pertinent direction for a coil-type specimen) is similar to that of Nb₃Sn-Cu. Based on the thermal contraction of G-10CR and G-11CR plate,^{4,5} the thermal contraction of G-10 plate tubes may be slightly closer to that of a Nb₃Sn-Cu wire than the G-11 plate tubes that were measured here. It is possible that the difference in the circumferential thermal contraction between the warp and fill directions will result in a spatial variation of the strain state of the Nb₃Sn-Cu coil sample, but the I_c data indicate that this effect is not significant.

The plate tubes present some practical disadvantages. First of all, machining a specimen holder from plate stock is considerably more difficult than from tube stock. Also, the length of a plate tube is limited by the thickness of the available plate stock. This, in turn, limits the length of the superconductor specimen for a given coil diameter and pitch. Furthermore, short specimen holders are often incompatible with existing I_c test fixtures. Satisfactory specimen holders of greater length could perhaps be constructed by bonding a series of short plate tubes together. This technique might require an alignment of the warp and fill fibers between individual tube sections because of the anisotropic radial thermal contraction of plate tubes.

CONCLUSIONS

For I_c measurements, fiberglass-epoxy composites are suitable specimen holder materials. However, the design of the specimen holder should take into account the anisotropic nature of the material and the resulting variability in thermal contraction. These characteristics of the material can result in large variations in the measured I_c for specimens mounted on holders of different designs. A cylinder or tube can be machined from a thick fiberglass-epoxy plate with the axis of the cylinder perpendicular to the surface of the plate. This type of tube (plate tube) has a thermal contraction that is relatively independent of its dimensions and that is similar to that of a Nb_3Sn -Cu specimen. Alternatively, the specimen holder can be made from tube stock and machined to a wall-to-radius ratio that results in a thermal contraction that closely matches that of the Nb_3Sn -Cu specimen.

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