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“Critical Current Metrology for Nb₃Sn Conductor Development”

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Introduction

NIST has played a key role in many of the one-on-one, domestic, and international interlaboratory comparisons of measurements on superconductors. The history of interlaboratory comparisons of measurements on superconductors tells us that careful measurement methods are needed to obtain consistent results [1-10]. Inconsistent results can lead to many problems including: a mistrust of the results of others, unfair advantages in commerce, and erroneous feedback in the optimization of conductor performance. NIST has experience in many interlaboratory comparisons; a long-term commitment to measurement accuracy; and independent, third-party laboratory status. The principal investigator's direct involvement in the measurements and daily supervision of sample mounting is the unique situation that has allowed important discoveries and evolution of our capabilities over the last 30 years. The principal investigator's research and metrology has helped to improve the accuracy of critical-current (I_c) measurements in laboratories throughout the world. As conductors continue to improve and design limits are tested, the continuation of the long-term commitment to measurement accuracy could be vitally important to the success of new conductor development programs.

It is extremely important to the U.S. wire manufacturers to get accurate (high certainty) I_c measurements in order to optimize conductor performance. The optimization requires the adjustment of several fabrication parameters (such as reaction time, reaction temperature, conductor design, doping, diffusion barrier, Cu to non-Cu ratio, and twist pitch) based on the I_c measurement of the conductor. If the I_c measurements are made with high variability, it may be unclear whether or not the parameters are being adjusted in the optimal direction or whether or not the conductor meets the target specification. Our metrology is vital to the U.S. wire manufacturers in the highly competitive international arena and to meet the aggressive performance goals.

The latest high-performance Nb_3Sn wires are being designed with higher current densities, larger effective filament diameter, less Cu stabilizer, and, in some cases, larger wire diameters than ever before [11-12]. In addition, some of the conductor designs and heat treatments cause the residual resistivity ratio (RRR , ratio of room temperature resistivity to the resistivity at 20 K) of the stabilizer to be less than 20. These parameters are pushing the conductors towards less intrinsic stability, into a region we call marginally stable [13-17]. These

parameters also create a whole series of challenges for routine I_c testing on short-samples, even when tested with the sample immersed in liquid helium.

High-current, variable-temperature I_c measurements are even more difficult than those made in liquid helium because the sample is only cooled by flowing helium gas. Providing accurate I_c results under these conditions requires a complex system that provide adequate cooling as well as uniform sample temperature. We have been make variable-temperature measurements for about 15 years, but we started to design the first high-current (at least 500 A), variable-temperature, variable-strain apparatus in late 2006. Our first critical-current measurements as a function of strain, temperature, and magnetic field, $I_c(B, T, \epsilon)$, in a new single, *unified* apparatus (full matrix characterization) were made in the summer of 2008. This is the only such facility in the U.S. and it has some unique components that are not duplicated anywhere in the world. The compounding of all three variables (H , T , ϵ) makes an already labor and time intensive characterization very formidable; however, the results cannot be generated any other way and are needed to answer key questions about strain and temperature safety margins and about the reliability of using scaling laws based on small data sets to predict performance. In the future, this new apparatus will allow NIST to create a database on strands that would benefit U.S. superconductor wire manufacturers, national research laboratories, and programs using superconductor strands such as HEP and International Thermonuclear Experimental Reactor (ITER).

Key Results of the program during FY'07-10

In the previous period of this program, we had extensive collaborations with industries and national laboratories. Work planning of our NIST superconductivity research program was determined in consultation with the wire manufacturers and a number of research collaborators within the DOE/HEP, LARP, SBIRs, and DOE/OFES communities. Direct feedback was given to the wire manufacturers within each project and then disseminated to the general wire-development community. A summary of key results obtained for the HEP program during the period FY'07-10 is given below.

1. New variable strain/temperature/magnetic-field apparatus:

We have an ongoing research program for characterization of superconductor composite strands, the principal output of which is sensitive measurements of critical current I_c over a broad range of the essential parameters: longitudinal strain ε , temperature T , and magnetic field B . The program features a new apparatus for integrated measurement of $I_c(B, T, \varepsilon)$ on the same, long-conductor sample without remounting. We finished the construction of the unified apparatus in close collaboration with Najib Cheggour. The unified apparatus combines world-class capabilities in variable-strain and variable-temperature measurements. The variable-temperature components of the apparatus were commissioned, calibrated, tested, and used.

The new apparatus allows us to measure the strand in compression and tension to cover a range of applied strain typically from -1% to $+1\%$, based on Cheggour's original design [18, 19] ("Durham probe" using a "Walter's spring" technique [20]). The variable temperature design utilizes our high-rate helium gas flow and gas temperature pre-regulation approach that was developed at NIST. This unique approach is expected to be one of the most reliable for conducting high-current measurements on low temperature superconductors. The new apparatus has reached currents above 700 A through a gas cooled sample. This apparatus was designed to fit in our existing 16.5 T/52 mm bore superconducting magnet.

Figure 1 shows the upper part of the new sample probe. A worm gear is used to torque the top of the spring while the bottom of the CuBe spring is held fixed. The angular deflection of the spring (which has an operating range of about $\pm 55^\circ$) is read through one of the windows at room temperature. Figure 2 show a T-section spring with a helical Nb₃Sn sample soldered to the spring. The spring has four active turns with solid ends. The current contacts are made at each end of the spring. Three pairs of voltage taps are located on the sample along the center three turns of the spring. Each voltage tap pair covers one turn of the spring and has a separation of about 8 cm. We are typically able to measure critical currents over a range of electric-field criteria from 0.01 to 1.0 $\mu\text{V}/\text{cm}$ (1 to 100 $\mu\text{V}/\text{m}$). We chose to use three pairs of voltage taps in order to improve the statistics of our measurements. This is especially important for our determinations of irreversible strain limits because we were able to obtain three separate determinations from each specimen. Our data acquisition system uses three nano-voltmeters to measure these three taps simultaneously.

Figure 1. Photograph of the upper part of the new high-current apparatus constructed at NIST to measure the critical-current dependence on strain, temperature and magnetic field. The worm-wheel that torques the spring can be seen through one of the windows.

Figure 2. Photograph of a T-section, CuBe spring with a helical sample soldered to the spring. Three pairs of voltage taps cover the three central turns of the spring. The current contacts are made at each end of the spring.

The CuBe spring, Cu current contacts (Cu lugs), and solder tabs form a modular sample mount (see Fig. 3). The Cu lugs are mechanically held to the CuBe spring with sets of pins, screws, and solder. This sample mount is soldered and instrumented before it is installed on the apparatus. The solder tabs are made of Cu strip and a number of layers of YBCO coated conductors. The tabs are imbedded in the Cu lugs and the current transfers from the YBCO to the helical sample over the length of the Cu lugs. The portion of the tab that extends past the Cu lug

allows for the final current connection to be made between the solder tabs and bus bar in the sample probe.

The sample region of the probe is shown in Fig. 4. Top and bottom Cu terminals nest around the sample mount. Grooves in the terminals allow the bus bar to solder tab connections to be made without significant heat transfer to the Cu terminal or lugs. Thermometers and heaters are mounted on each terminal. Sets of pins (3 mm diameter) and screws make the mechanical connections between the terminals and the Cu lugs. The top terminal is pinned and screwed to the central torque shaft that is connected to the worm wheel at the top of the sample probe (see Fig. 1). Another set of pins and screws are used to attach the bottom terminal to a stainless steel can that fits over the sample and couples the torque between the central shaft and the outer structure of the probe. These sets of pins transmit/react the torque that is applied to the spring.

The sample probe is inserted into a re-entrant Dewar, which is in-turn inserted into a Dewar with a superconducting magnet. A schematic diagram of this arrangement and helium flow paths are shown in Fig. 5. The re-entrant Dewar has vacuum, super-insulation, and a

Figure 3. Photograph of CuBe spring (7.2 cm), Cu current contacts (Cu lugs), solder tabs, and helical sample that form a modular sample mount (shown demounted from probe).

Figure 4. Photographs of sample mounted to end of the probe with two orientations to illustrate how the solder tabs nest with the top and bottom terminals and where the heaters and thermometers are located on the terminals.

liquid helium transfer tube. The transfer tube allows liquid helium to flow from the magnet Dewar to the middle of the re-entrant Dewar. The rate of helium flow is controlled by the impedance of a needle valve and the pressure difference between the two Dewars. The variable temperature system has four separate control loops that work together to provide the necessary cooling power for the sample and the sample current leads. Control loop #4 is the pressure control of the magnet Dewar, which determines the helium flow rate and the total power of the heaters in the other three control loops. The computer monitors the total heater power and slowly adjusts the pressure set-point to attempt to achieve a certain target heater power. The total heater power target depends on the intensity of sample current. Control loop #3 is the heater and thermometer on the pre-regulator. The pre-regulator attempts to heat the helium so that the gas flowing over the sample provides just the right amount of cooling power for the sample to reach

Figure 5. Schematic diagram of variable temperature apparatus that shows the arrangement of components.

a certain temperature set-point. This amount of cooling power again depends on the level of sample current. The computer monitors the heater power of control loop #1 (above the sample, which typically has the least amount of cooling) and slowly adjusts the temperature set-point to attempt to achieve a certain target heater power for control loop #1. Notice that the temperature set-point varies with time and is typically less than the sample temperature. Control loop #2 is below the sample and the helium gas flows over this control loop before it flows over the sample. Then the gas flows over control loop #1 that is above the sample. There is a small amount of gas that does not flow directly over heater #2 and the sample before it flows over control loop #1. This bypass flow provides a little more cooling for control loop #1 and allows it to control to the same temperature as control loop #2 even with small cooling powers. All of the gas flow continues up the center of the sample probe and out through the vapor-cooled sample-current leads.

There are many long-time constant interactions between the four control loops and the computer program needs to keep the system from going into oscillation. The reason for the four control loops will not become apparent to the reader until we examine the protocol necessary to achieve the “correct” critical-current measurements under flowing helium gas conditions to high current levels. We made comparisons between measurements in pressurized liquid helium at 5 K and flowing helium gas at 5 K to help determine the necessary protocol for accurate/“correct” variable temperature measurements in gas environment. The four control loops and the protocol attempt to create a relatively uniform temperature over the length of the sample and some relationship between the average thermometer temperature and the average sample temperature.

We have extensive experience making variable-temperature critical-current measurements; however, we knew that the variable temperature operation of the unified rig would be more difficult than our other variable temperature cryostats because of the compromises in design that we had to make to incorporate variable-strain capability. Some of the design compromises were: a smaller diameter working space, reduced thermal mass and cooling surfaces of the current contacts, a longer sample length between the current contacts, larger distances between thermometers and the sample, and an off-balance of cooling power to each contact. As we expected, the new cryostat is much more sensitive to protocol and we will need to make further hardware and software changes to meet these requirements. Despite these challenges, we made measurements to currents above 750 A with the sample in gas cooling at

5 K, which is a very impressive result. The results of the protocol studies indicate that the effective sample temperature, with gas cooling and the minimum sample heater power, is only sub-cooled by about 0.05 K.

2. Comparison of measurements in liquid and gas at 5 K:

The re-entrant Dewar can be sealed, filled with liquid helium, pressurized, and the liquid heated to 5 K. This allows us to measure the sample at 5 K with the sample immersed in liquid helium, which we expect will give us the “correct” critical current ($I_{c \text{ liq}}$). In these conditions, we expect the minimum difference between the temperature measured by the thermometers and the sample temperature. The high-gas flow design of the apparatus also allows us to make measurements at 5 K with the sample cooled by helium gas flow. Based on our past experience, we expect that the value of critical current measured in flowing helium gas ($I_{c \text{ gas}}$) will depend on the amount of heater power. Higher heater power near the sample will be correlated with more sub-cooling of the sample relative to the thermometers and a higher measured $I_{c \text{ gas}}$. So we conducted a systematic study of measured $I_{c \text{ gas}}$ with different heater powers. This type of study only has direct implications for our specific apparatus, but it should give us an indication of our temperature uncertainty and the protocol (heater settings, gas flow rates ... etc) that gives us the least uncertainty in sample temperature. All of the I_c shown in this report used an E_c criteria of 0.1 $\mu\text{V}/\text{cm}$ (10 $\mu\text{V}/\text{m}$). This study showed that the ΔI_c (%) defined as $(I_{c \text{ gas}} - I_{c \text{ liq}})/I_{c \text{ liq}}$ correlates with the heater power of control loop #2 ($H2$ power). ΔI_c did not correlate as well with $H1$ power and there was very little, if any, correlation with the pre-regulator heater power. It is postulated that the $H2$ power is closely related to the amount that the flowing helium gas is sub-cooled relative to the thermometer considering that immediately after flowing over control loop #2, the gas flows over the sample. More specifically, the minimum $H2$ ($H2\text{-min}$) heater power measured during the time that the sample current was near I_c is the heater power of interest. Figure 6 shows ΔI_c (%) as a function of $H2\text{-min}$ for various magnetic fields where the I_c varied from 12 to 764 A. An applied strain of -0.36% , on a 0.7 mm diameter, high-performance Nb_3Sn LARP conductor, was selected so that a low value of I_c could be achieved at our highest magnetic field of 16 T. At the lowest $H2\text{-min}$ for each magnetic field, ΔI_c (%) was less than the larger of 2 % and 2 A. ΔI_c (%) increases with increasing $H2\text{-min}$ with an approximately linear relationship. $I_{c \text{ gas}}$ was the closest to the correct $I_{c \text{ liq}}$ when $H2\text{-min}$ was closest to zero. Obviously, we tried to

make measurements at the lowest possible $H2$ powers without losing sample temperature control; however, $H1$ power would go to zero before $H2$, especially at the higher currents. The lowest achievable powers, without losing temperature control, were higher for the larger I_c . The approximate linear relationship has an x -axis intercept, $\Delta I_c (\%) = 0$, between 0 and 0.1 W for the higher currents. The x -axis intercept for the current below about 250 A was near 0 W. The different slopes of these data at different magnetic fields/critical currents are expected to be due to the different sensitivities of $\Delta I_c (\%)$ to temperature.

We have postulated that the dependence of ΔI_c on $H2$ -min power is due to sub-cooling of the sample. If this is true, we should not look at ΔI_c versus $H2$ -min power, but at the *apparent temperature error* as a function of $H2$ -min power. The *apparent temperature error* is calculated using ΔI_c and the measured temperature dependence of $I_{c \text{ liq}}$. This should represent the

Figure 6. $\Delta I_c (\%)$ defined as $(I_{c \text{ gas}} - I_{c \text{ liq}})/I_{c \text{ liq}}$ versus the minimum heater power of control loop #2 ($H2$ -min (W)) at various magnetic fields where I_c varies from 12 to 764 A at 5 K for a high-performance RRP Nb_3Sn wire. Smaller $H2$ heater power achieves a better match of the temperature of the helium gas to that of the sample (see Fig. 7).

mechanism that changes I_c and thus, *apparent temperature error* should be the intrinsic factor that does not depend on the sensitivity of I_c to temperature at the particular magnetic field, temperature, or strain. Figure 7 represents the *apparent temperature error* as a function of $H2-min$ power for the same data set as shown in Fig. 6. For I_c below 150 A, the points for various I_c 's fall on the same line that has a zero *apparent temperature error* intercept that is about -0.01 to -0.02 W. This intercept moves to positive values for I_c above 250 A; however, the slopes are very similar over the whole range of measured I_c . This sub-cooling relationship should not be thought of as a function of magnetic field, but as a function of critical current. For this range of current, the *apparent temperature error* is about 0.05 K or less for the minimum achievable $H2-min$ power values. It may be possible to make a first order correction to the measured $I_{c\text{ gas}}$ using this relationship to reduce the bias further. We intend to repeat this comparison of liquid and gas measurements on each sample we measure until more is known about how universal these

Figure 7. *Apparent temperature error* versus the minimum heater power of control loop #2 ($H2-min$ (W)) at various magnetic fields where I_c varies from 12 to 764 A at 5 K for a high-performance RRP Nb₃Sn wire.

observations are. One general conclusion we can make is that at low currents (below 150 A) a heater power as little as 0.1 W can create an *apparent temperature error* of more than 0.1 K.

We could only do a comparison of $I_{c \text{ gas}}$ and $I_{c \text{ liq}}$ at 5 K, but this leaves the question open as to whether the same protocol leads to different results at higher temperatures where the heat capacity of the helium gas (linear vs. temperature), sample ($\propto T^3$ in metal), and test fixture is higher. Thus, we studied the measured $I_{c \text{ gas}}$ at 8 K as a function of $H2\text{-min}$ power. Figure 8 shows the *apparent temperature error* as a function of $H2\text{-min}$ power at 8 K for various magnetic fields where the I_c varied from 14 to 591 A at an applied strain of 0.0 %. There is no way to know the “correct” I_c , so the value of $I_{c \text{ gas}}$ measured at the lowest $H2\text{-min}$ power was used instead to calculate the *apparent temperature error*. This caused data to shift arbitrarily, but

Figure 8. *Apparent temperature error* versus the minimum heater power of control loop #2 ($H2\text{-min}$ (W)) at various magnetic fields where I_c varies from 14 to 591 A at 8 K for a high-performance RRP Nb_3Sn wire. Since measurements at 8 K could only be done in flowing gas, $I_{c \text{ gas}}$ measured at the lowest $H2\text{-min}$ power was used instead of $I_{c \text{ liq}}$ to calculate the *apparent temperature error*.

allowed us to see that the slopes of this relationship are very similar to the slopes measured at 5 K. This may be because the protocol is based on a heater power that removes the heat capacity effect. Thus, we expect that the protocol to remain similar for a reasonable range of temperatures.

When we were not studying the change in measured $I_{c\text{ gas}}$ as a function of *H2-min* power, we attempted to use the protocol that resulted in the minimum *H1* heater power, which was essentially the minimum *H2* heater power, without losing sample temperature control during a *V-I* curve. Otherwise, the protocol was the same during all measurements. The details regarding the protocol for control loops #3 and #4 are explained below. Control loops #1 and #2 attempt to hold their respective thermals at a certain temperature. Control loop #3 (pre-regulator) does not care about the sample temperature set-point; the computer makes appropriate changes in the temperature set-point of the pre-regulator in order to achieve a certain *H1* heater power target. Similarly, control loop #4 also does not care what the sample temperature set-point is; the computer makes appropriate changes in the magnet Dewar pressure set-point in order to achieve a certain total heater power target (the sum of *H1*, *H2* and the pre-regulator powers). The interactions between these four control loops is very complex and we continue to attempt to improve the control software to avoid long-time constant oscillations and loss of temperature control especially after making temperature steps of 1 K or more.

The function of the pre-regulator (control loop #3) is to reduce the temperature gradients along the sample and allow for better temperature control and higher gas flow rates without excess cooling power. The pre-regulator is also situated to help intercept some of the heat flowing down and that generated in the sample current leads. So, as the *V-I* curve is measured, most of the heat from the sample current will replace some or all of the pre-regulator heater power. In other words, during a *V-I* curve measurement, we attempt to efficiently use the heat from the sample current to pre-heat the helium. The total power target of control loop #4 is set according to the expected sample current in order to attempt to keep the pre-regulator heater power above zero during a *V-I* curve measurement. The higher target powers for the higher currents means that the gas flow rate over the sample will be higher for the higher sample currents. This also minimizes change in the heat flow to the sample during the curve.

3. $I_c(B, T, \epsilon)$ data on high-performance Nb₃Sn LARP conductor:

The compounding of all three variables (B, T, ϵ) makes an already labor and time intensive characterization very formidable; however, the results cannot be generated any other way and are needed to answer key questions about strain and temperature safety margins of HEP magnets and about the reliability of using scaling laws based on small data sets to predict performance. Full characterization of a single sample is expected to require about two months of intensive work and significant consumption of liquid helium. Currently, the community perception is that every Nb₃Sn conductor needs to be fully characterized due to the lack of consensus on unified scaling and due to the larger extrapolations needed for some new systems. We hope that our future data will be a basis for consensus on unified scaling and/or define the limits to scaling. Unified scaling is expected to allow for more precise estimations of conductor performance over a wide range of parameters using a more limited and cost effective data set.

Some of the $I_c(B, T, \epsilon)$ measurements on a 0.7 mm diameter, high-performance RRP Nb₃Sn sample for LARP are shown in Fig. 9, 10, and 11. Applied strain started at 0 %, and then was stepped to a maximum value of about 0.3 %, and then brought back to -0.1 % and stepped down to about -0.7 %. The maximum applied tensile strain of 0.3 % was selected to keep the sample within the reversible region. These high-performance wires have an irreversible strain limit that is just past where the critical current peaks with strain. At each strain, temperature was set and magnetic field was swept through the range where I_c could be measured. Then a new temperature was set and magnetic field swept again. This process was repeated until all of the temperature set-points were completed for a given strain.

The 5 K data shown in Fig. 9 were taken with the sample in flowing helium gas, which was easier and faster than sealing the re-entrant Dewar, filling it with liquid helium, pressurizing, and heating the liquid to 5 K. In general, only the measurements at 4 K were done with the sample immersed in liquid helium. There are 86 I_c points on this plot (Fig. 9). We typically attempted to take I_c data to current above 600 A at 5 K and these magnetic fields. Figure 10 shows 98 I_c points at 8 K. As with the measurements at 5 K, the sample was typically stable enough for sample currents up to about 600 A at these higher magnetic fields. In contrast, Fig. 11 shows 85 I_c points at 12 K where the sample stability limited the current to about 400 to 500 A. The sample did not seem to be as stable at these lower magnetic fields (<3.5 T).

We also took $I_c(B, T, \epsilon)$ measurements at a couple strain points and two temperatures below 4 K (2.4 and 3.3 K) to get some additional information on temperature scaling. The existing temperature scaling models are all able to fit the temperature data above 4 K, but differences seem to appear when data below 4 K are added. We are continuing to study the scaling relationships using these data.

Figure 9. Critical current as a function of applied strain at various magnetic fields and 5 K in flowing helium gas for a high-performance RRP Nb₃Sn wire.

It takes about 8 hours to complete the measurements as a function of temperature and magnetic field at one strain point. We made measurements at 11 different strain points and we repeated the measurements again at zero applied strain after the -0.7% strain point. The comparison measurements in liquid and gas at 5 K were done at applied strains of 0% and -0.36% , which took about two days at each strain point. In all, it took more than three weeks of data acquisition to get about 1400 critical-current values on one Nb_3Sn specimen in a three-dimensional matrix of 11 strain settings (-0.7% to $+0.3\%$ applied strain), eight temperatures (4, 5, 6, 8, 10, 12, 14, and 15 K), and six to ten magnetic fields (up to 16 T).

Figure 10. Critical current as a function of applied strain at various magnetic fields and 8 K in flowing helium gas for a high-performance RRP Nb_3Sn wire.

Figure 11. Critical current as a function of applied strain at various magnetic fields and 12 K in flowing helium gas for a high-performance RRP Nb₃Sn wire.

4. $I_c(B, T, \epsilon)$ data on 10% SiC doped MgB₂ wire:

Some of the $I_c(B, T, \epsilon)$ measurements on a 0.84 mm diameter, 10 % SiC doped MgB₂ wire are shown in Fig. 12. The wire had seven filaments and a monel jacket. Only a few strain points are shown in Fig. 12 for clarity. Data were taken at six applied strain points from 0 to 0.5 %. All of these data in Fig. 12 were taken in flowing helium gas with a maximum current just over 300 A.

Figure 12. Critical current density as a function of applied field at various temperatures and strains in flowing helium gas for a MgB_2 wire. For clarity, only data at three strain points are shown.

We also performed a comparison of measurements in liquid and gaseous helium at 5 K on this MgB_2 wire. Figure 13 shows the *apparent temperature error* as a function of $H2\text{-min}$ power for various magnetic fields where the I_c varied from 1.9 to 220 A. These data points are very similar to those in Fig. 7 (Nb_3Sn) with currents below 230 A. This gives confidence that the *apparent temperature error* may be fairly universal as long as contact heating or sample inhomogeneity did not contribute to sample heating. The lower temperature sensitivity of I_c for MgB_2 compared to that for Nb_3Sn made the MgB_2 comparison at 5 K more sensitive to data scatter than to temperature fluctuations. Also, these MgB_2 measurements were made at an earlier stage of the data-acquisition-software development, and before a small hardware change were made to the gas flow path, so it is fairly remarkable or a coincidence that these results are so similar.

Figure 13. Apparent temperature error versus the minimum heater power of control loop #2 ($H2-min$ (W)) at various magnetic fields where I_c varies from 1.9 to 220 A at 5 K for a MgB_2 wire.

5. Walter's spring calibration:

The Walter's springs were calibrated to measure the strain-angle relationship. Several strain gauges were glued to the outer surface of each spring. To check the effect of temperature, the calibration was performed at room temperature, 76 K and 4 K. The longitudinal strain varies linearly as a function of the angle of twist for both compressive and tensile strain regimes. As expected from design calculations, strain is reversible within the strain window from -0.7% to $+0.7\%$ for the rectangular-section spring, and within the strain window from -1% to $+1\%$ for the T-section spring. Furthermore, calibrations performed at different temperatures show that the calibration factor is temperature-independent within 3 % between room temperature and 4 K, and within 1 % between 76 K and 4 K.

We wrote a data acquisition and analysis program for the spring calibration. The program initiates simultaneous measurements for up to five strain gages and records and displays these values. The precise measurements allowed for comparisons of the strain in different portions of the spring, through many strain cycles, and at different temperatures.

6. Magnetic field calibration:

We had been using the factory calibration of our 16.5/18.5 T magnet system and we needed to verify this calibration using NMR probes. The lower field homogeneity of this magnet (compared to some of our other magnets) made an NMR calibration more difficult, especially at the higher fields. The high-field calibrations agreed with the factory calibration to within about 0.1 %. We also determined the magnetic field hysteresis with sweep direction and the low-field non-linearity of the field-current relationship.

We wrote a LabVIEW data acquisition and analysis program for magnetic field calibration using NMR probes from 0.05 to 13.7 T. This calibration is an ideal application for a LabVIEW program since it can process and display averaged waveforms better than a digital-processing oscilloscope. Figures 14 and 15 show screen captures of the computer displays at 2.05 and 13.7 T respectively. The main curve is a 30 wavelength average of the NMR signal, using the zero-crossings of the triangular field-modulation signal. There is a slight asymmetry in the NMR signal with the sweep direction of the field modulation. The signal-to-noise ratio was best at 2.05 T (~0.2 V peak-to-peak NMR signal) and worst at 13.7 T (~0.03 V peak-to-peak NMR signal). The display is used to find the magnetic center and to manually fine-tune the resonance signal at each field set point.

7. Critical current measurements:

We are developing software for the automation of $I_c(H, T, \epsilon)$ measurements. Some level of automation is absolutely essential for this research and it must be implemented early in the project. Further development of more advanced automation is expected to help maintain data reliability and increase the number of fully characterized samples per year. The acquisition needs to be robust to run through a sequence of measurements with minimum human input. The computer programs are expected to be very specialized for our systems and evolve over the next few years.

Figure 14. Screen capture of NMR calibration program with field setting at 2.05 T. The main plot is a digitally averaged NMR waveform that has resonance near the time points marked 300, 800, and 1300.

Figure 15. Screen capture of NMR calibration program with field setting at 13.7 T. The main plot is a digitally averaged NMR waveform that has resonance near the time points marked 300, 800, and 1300.

We have used the Basic language program for instrument control and data acquisition and analysis for about 25 years. For our variable-temperature measurements, we now use two computers on separate buses to communicate with about 25 instruments (voltmeters, scanners, temperature controllers, voltage sources, and pressure gauges). These instruments allow us to control the magnetic field, sample temperature, sample current, sample current ramp rate, and system pressures (gas flow rates or liquid temperature). They are also used to monitor sample voltages, sample current, system pressures, gas flow rate, heater powers, contact resistances, voltage drop along various segments of the current path, sample quench, liquid helium levels, and temperatures of various parts of the apparatus. Over the past years, we have had fully autonomous and robust programs that could make a sequence of measurements without human assistance. In recent years, these programs have not been robust enough to handle the new challenges of higher currents, measurements in a gas flow environment, and marginally stable samples. The Basic programs are also not very user friendly. So, we have decided to create LabVIEW programs that will run on one computer with fewer instruments, new predictive algorithms, and new control signals for more robust operation. We also need the program to use multiple acquisition modalities to accommodate various sample properties and variable-temperature conditions while maintaining accuracy and efficiency.

Figure 16 shows a screen capture of our critical-current data acquisition and analysis program. There are 12 different tabs (screens) that include initialization, run matrix (strain, temperature, and field points), displays, results, run time data, advanced controls, system state, and stability stats. The plot on the left shows $I_c(\epsilon)$ at 12 T with loaded and unloaded strain points for each of the three voltage taps. On the right are full-logarithmic plots of $E-I$ for each of the three regions of the sample. The lower plot shows the raw $E-I$ points and the upper plot shows critical current values at various criteria. These run-time displays give the operator feedback on the relative consistency of the three sample regions, the location of the peak critical current, and where the strain effect becomes irreversible. Other run-time displays show sample voltage and current readings as a function of time and voltage drops over various portions of the sample current path including each end of the sample and the current contacts to the sample.

Figure 16: Screen capture of the front panel of LabVIEW critical-current data acquisition and analysis program. There are 12 different tabs (screens) that include initialization, run matrix (strain, temperature, field), displays, results, run time data, advanced controls, system state, and stability stats.

8. Magnetoresistance of resistive thermometers:

We developed new software to measure the magnetoresistance of resistive thermometers using a capacitive thermometer. We have done this type of measurement before with Basic software, but again, we wanted to make it more user friendly so that others in our project could use this software. This experiment also gave us more experience with variable temperature operation that is highly relevant to the unified apparatus. A number of the sub-programs will be incorporated into the critical-current software.

Measurements were made at various temperatures. The magnetic field was swept from 0 to 16 T, and then back to 0 T again. We used fixed temperatures of 4, 4.5, 5, 6, 7, 8, 9, 10, 11, 12, 14, 16, 18, 20, 25, 30, 35, 40 and 76 K. Measurements at 4, 4.5, and 5 K were done in liquid

helium and those at 76 K were done in liquid nitrogen. Our re-entrant Dewar can be filled with a liquid cryogen, pressurized, and heated to change the temperature of the cryogen.

Figure 17 shows a screen capture of the thermometer magnetoresistance data acquisition program. The two plots are the measured temperature of two thermometers as a function of time while ramping the magnetic field up and down. The magnetic field is zero at each end of the curves and reaches 16 T near the center of the plots. The field is held at 16 T for one minute. The true temperature is fairly constant, near 6 K, for the entire curve. The apparent change in temperature is about 60 mK peak-to-peak for the top thermometer and is about 120 mK peak-to-peak for the bottom thermometer.

Figure 17: Screen capture of the front panel of the LabVIEW thermometer magnetoresistance data acquisition program. The true temperature is 6.00 K and fairly constant for the entire curve as the magnetic field is swept from 0 to 16 T, and then back to 0 T.

9. Method for determination of the irreversible strain limit of Nb₃Sn wires:

We have developed a more rigorous method for the determination of the irreversible strain limit of Nb₃Sn wires. The critical current is measured as a function of applied strain, $I_c(\epsilon)$, at one magnetic field and 4 K. A typical data set is shown in Fig. 18 for a high-performance Nb₃Sn wire made by the PIT process [21] at an electric-field criterion, E_c , of 0.1 $\mu\text{V}/\text{cm}$. These data were taken on a reference sample that was not reinforced. The sample is loaded and (partially) unloaded several times to determine ϵ_{irr} , which is defined as the maximum loaded strain where the I_c is still reversible. The periodic unloading is done to determine if I_c is still reversible by comparing the unloaded I_c to a previous, lower-strain measurement of I_c . All of the loaded points are indicated by the solid symbols starting from zero applied strain. All of the unloaded points are indicated by open symbols. The 14th loaded point is labeled as point A. After point A, the strain is reduced by a certain amount and point A' is measured. The strain is then increased to point B and again reduced to point B' and so forth. The amount that the strain is

Figure 18: Critical current versus applied strain for a 0.5 mm diameter PIT Nb₃Sn wire at 12 T and 4 K. Loaded points are plotted with solid symbols and unloaded points are plotted with open symbols. Some of the loaded and unloaded pairs of points are labeled with unprimed and primed letters respectively.

partially unloaded is about 0.09 % (this corresponds to an angle change of 5° for the spring). There are trade-offs with the unload amount, but this amount is large enough to give a good signal and is low enough to minimize the work-hardening of the sample.

Six polynomials, second degree through seventh degree, are each fit to all of the loaded data points. The highest order polynomial for which the last term is statistically significant is used to calculate percent residuals for both loaded and unloaded points. The percent residuals are defined as $100(I_c - I_{c \text{ predicted}})/I_{c \text{ predicted}}$. The residuals for loaded and unloaded points represent the deviation from the model fit that was based on only loaded data. Figure 19 displays percent residuals for both loaded (solid circles) and unloaded (open circles) points. The loaded curve in Fig. 18 has two components; an intrinsic dependence of $I_c(\epsilon)$, plus an I_c degradation that increases with strain beyond some strain value. The second component does not necessarily increase smoothly with strain because of the nature of fractures. This can cause problems for the fit at the high strain points as can be seen in Fig. 19. We typically limit this fit of the loaded

Figure 19: A seventh degree polynomial was fit to all of the loaded strain points. The percentage residuals from this fit are plotted versus applied strain. The plot shows that it is difficult to fit the entire loaded curve.

points to strain point Q and below since we do not need to fit the loaded points past the highest unloaded point V'. However, we show the complete fit to demonstrate the exaggerated influence of the high strain points on the overall fit. Since we measure I_c for three different segments of each sample and at three different E_c criteria (0.01, 0.1 and 1.0 $\mu\text{V}/\text{cm}$), we often have some data that were taken to higher strains than we need. Using this first fit, we attempt to find the lowest unloaded strain point where the residual is less than -5% , which is point V' in this case.

We then trim and fit the loaded data set to this strain point, which is point Q and below in this case. The order of the polynomial fit was 4th for the trimmed and 7th for untrimmed data. Figure 20 displays percent residuals for both loaded and unloaded points for the 4th order polynomial using trimmed data. The residuals for the loaded data are very small except for points M and N. Apparently there was some larger than expected change in I_c between these two points and the smooth fit could not accommodate the abrupt change. There is also an abrupt change in the residuals for the unloaded curve between points M' and N'. The residual of point M'

Figure 20: A fourth degree polynomial was fit to a trimmed subset of the loaded strain points. The percentage residuals from this fit are plotted versus applied strain. The plot shows the degradation of unloaded I_c as the load is increased.

($\epsilon=0.49\%$) is very close to zero, which indicates that I_c at the unloaded point M' matches I_c at the loaded point H. Thus, the I_c was still reversible at the strain of M ($\epsilon=0.58\%$). In contrast, the residual of point N' ($\epsilon=0.51\%$) is about -0.7% , which indicates that I_c at point N' is lower than I_c at point I by 0.7% . Therefore, the I_c was not completely reversible at the strain of N ($\epsilon=0.60\%$) and there was permanent I_c degradation when the strain went to 0.60% . This type of analysis would determine the ϵ_{irr} to be about 0.58% . In general, we do not do a point by point comparison because these data have noise, we want to interpolate between points, and we might not have a loaded point at exactly the same strain as an unloaded point.

To do a general analysis that takes into account the strain that each unloaded point was unloaded from, we re-plot the unloaded residuals (unloaded y-values) at the corresponding loaded strains (loaded x-values). This essentially shifts the unloaded points by the amount that the strain was unloaded. Figure 21 shows the resulting points that are the original unloaded residuals (labeled unshifted) and the *shifted unloaded* residuals. In other words, point Q in Fig.

Figure 21: Plot of percent residuals versus applied strain for the unloaded points using the original strain scale (open circles) and the shift strain scale (solid circles) corresponding to the loaded data. The irreversible strain limit can be determined by fitting and interpolating the *shifted unloaded* residuals, the solid points.

21 is plotted at the same strain (loaded x -value) as point Q in Fig. 20, but the y -value of point Q in Fig. 21 is plotted as y -value of point Q' in Fig. 20. This maps the I_c degradation to the corresponding loaded strain values, thus these *shifted unloaded* data become the I_c degradation curve. Now we can fit and interpolate these *shifted unloaded* data to get the ϵ_{irr} . Because of noise in the data points near zero, we fit the two *shifted unloaded* points surrounding -1% I_c degradation to back extrapolate to zero I_c degradation to determine ϵ_{irr} . In this case we get $\epsilon_{irr}=0.568\%$. An extra digit is given here to facilitate comparisons. We can also determine *critical strains*, ϵ_c , at various percentage I_c degradations such as $\epsilon_c=0.635, 0.694$, and 0.723% at I_c degradations of $-1, -2$, and -5% respectively. These ϵ_c values indicate the shape of the degradation curve with increasing strain. The shape of the degradation curve shown in Fig. 21 is in the middle of what we have observed for a number of samples; some curves are broader (indicating more uncoupled filament fracture) and some are sharper (indicating more coupled filament fracture). Another use for the -1 and -2% ϵ_c values is a check of the extrapolated ϵ_{irr} . Because of I_c measurement noise and actual variability in the incremental amount of I_c degradation with increasing strain, the back extrapolation can lead to an erroneous ϵ_{irr} . In the case where the back extrapolated ϵ_{irr} using data around -1% and the back extrapolation using ϵ_c at -1% and -2% , define this as $\epsilon_{c,0}$, are somewhat different, the later has proven to be more reliable. In this case, $\epsilon_{c,0}=0.576\%$, which is only 0.008% higher than ϵ_{irr} .

The $I_c(\epsilon)$ curve goes through a peak at a strain ϵ_{max} that corresponds to the compressive pre-strain experienced by the Nb_3Sn filaments after cool-down from the heat-treatment temperature to 4 K . However, because the sample is soldered to a Cu-Be spring, the *absolute* values of ϵ_{max} and ϵ_{irr} are artificially higher due to the thermal expansion mismatch between Cu-Be and the sample. Whereas the values of ϵ_{max} and ϵ_{irr} quoted are not exact, those of the *intrinsic* irreversible strain limit ($\epsilon_{irr,0} = \epsilon_{irr} - \epsilon_{max}$) truly represent the amount of tensile strain that can be applied to the Nb_3Sn in the wire before filaments begin to fracture. The fit of the trimmed, loaded $I_c(\epsilon)$ is used to estimate the location of the peak in I_c . In the case of Fig. 18, $\epsilon_{max}=0.330\%$. So the $\epsilon_{irr,0}$ for this sample was 0.238% .

Also of note in Fig. 20 is that the unloaded residuals at strains below 0.35% are slightly positive. This slight enhancement of I_c is thought to be due to small decrease in the 3-dimensional strain that the superconductor experiences after the strain is cycled. In this case, the effect is only about 0.25% in I_c . The largest effect that we have observed during these

measurements is about +1 % in I_c . In rare cases, we have observed a slight negative effect on I_c . In the case shown in Fig. 20, slight enhancement of I_c seems to go away as the strain cycle continues, which may be due to subsequent enhancement of the loaded I_c 's that the unloaded values are being compared to.

The final plot of the *shifted unloaded* residuals versus intrinsic strain is all that is needed to determine ε_{irr} and the ε_c 's. Figure 22 is such a plot for a re-stacked-rod process (RRP®) Nb₃Sn wire that was doped with Ta [11]. The plot shows *shifted unloaded* residuals versus intrinsic strain for two samples of this wire, with three voltage taps on each sample. We now label the y-axis of this plot as I_c degradation. The average of the six determinations of $\varepsilon_{irr,0}$ is only 0.04 %. The small value of $\varepsilon_{irr,0}$ indicates that this wire has a very low tolerance to tensile strain. The I_c degradation curves are fairly consistent for all observations and they have a fairly sharp onset with strain just past the peak in $I_c(\varepsilon)$. Do not confuse this plot with $I_c(\varepsilon)$.

Figure 22: I_c degradation (*shifted unloaded* residuals) versus intrinsic strain for two samples, with three voltage taps on each sample. The average $\varepsilon_{irr,0}$ of these six determinations is only about 0.04 % for this Ta doped, RRP conductor.

Figure 23 is plot of I_c degradation (*shifted unloaded* residuals) versus intrinsic strain for an RRP Nb₃Sn wire that was doped with Ti instead of Ta. The I_c degradation curves in Fig. 23 show that the I_c was completely reversible with strains up to more than 0.2 % beyond the peak in $I_c(\epsilon)$, which is clearly different than the I_c degradation curves in Fig. 22. The average of the six determinations of $\epsilon_{irr,0}$ is 0.25 %, which is a significant improvement over the Ta doped wire made by the same manufacturer. These results on Nb₃Sn doping were presented at the Low Temperature Superconductor Workshop in November 2009 and were submitted for publication.

Preliminary studies were conducted on the effect of different acquisition methods for the determination of the irreversible strain limit. For example, is there any difference in irreversible strain limit if the amount that the strain is unloaded is changed? Unload strains of 0.05, 0.09, and 0.13 % (3°, 5°, and 7° changes in the deflection angle of the spring) were investigated. The range of $\epsilon_{irr,0}$ determined (average value 0.277 %) for these three different unloading amounts was only 0.009 %. The range of $\epsilon_{c,-5}$ was 0.040 % as expected because the smallest unloaded amount does not include as much of the I_c degradation as the largest unload. Even at $\epsilon_{c,-1}$ the range was only 0.011 %, so the value extrapolated back to zero percent degradation is fairly insensitive to the amount unloaded. In another study of a wire from the same batch as above, the unload strains of 0.09 %, 0.13 %, and back to the peak of $I_c(\epsilon)$ were used. In this case, the determined $\epsilon_{irr,0}$ values were 0.1 % to 0.2 % higher than the 0.277 % value determined above. These artificially high $\epsilon_{irr,0}$ values are likely caused by the work hardening of the sample caused by the inclusion of the large and constantly increasing strain swing back to the peak of $I_c(\epsilon)$. This strain swing also caused the unloaded I_c 's to be above the loaded curve by as much as 2 to 3 % indicating the accumulation of a positive three-dimensional strain effect. This interfered with the $\epsilon_{irr,0}$ determination more than anything.

Other studies have been performed using the measured n -value to determine the irreversible strain limit, which is the approach of some laboratories. Our studies show that, although the n -value is very sensitive to filament breakage, the n -value is noisy and is not a direct measure of changes in the current carrying capability of the wire. The n -value for some taps, on some wires, will actually continue to increase past the peak in $I_c(\epsilon)$. Using a lower I_c criterion of 0.01 $\mu\text{V}/\text{cm}$ can also increase the sensitivity to breakage, but it can be as noisy as the n -value. More research is needed in this area.

Figure 23: I_c degradation (*shifted unloaded residuals*) versus intrinsic strain for two samples, with three voltage taps on each sample. The average $\epsilon_{irr,0}$ of these six determinations is about 0.25 % for this Ti doped, RRP conductor.

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