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TPX Nb_3Sn Conductor Testing at LBL

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Abstract-

Two wire lengths (one from Supercon and one from IGC) were delivered for testing at the LBL Short-Sample Test Facility. Several samples of each wire-type were wound onto forms and reacted according to the requested prescriptions. Leads and voltage-tap wires were carefully attached after reaction according to standard LBL short-sample test procedures. Testing of some of the samples has been completed. Liquid helium immersion (4.2K) data was gathered over a limited range of magnetic fields (5-10T). Additional gas-cooled data was collected over a range of temperatures (1.8-14 K). Testing was interrupted when the test-magnet's persistent-switch-heater failed.

Good sample-to-sample and retest repeatability was observed for the 4.2K data when it was checked. Temperature measurements on the Supercon samples used CGR's and revealed a disappointing, non-repeatable (pressure-dependent) temperature offset for the gas-cooled measurements. We also observed a systematic dependence upon magnetic-field strength. Changing to a second CGR did not help. The IGC sample used a Cernox-type resistor which showed negligible magnetic and pressure dependencies.

Testing is expected to resume when the magnet is repaired.

I. Introduction

High magnetic field coils (B > 8T) that are designed for steady-state operation at elevated temperatures (T > 5K) require the use of conductors like Nb3Sn that are optimized for such operating conditions. One desires the conductor to be characterized adequately enough to accurately predict its performance near any desired operating points before one incurs the considerable expense of constructing and testing a realistic model.

Resistance measurements of "short-samples" of a proposed conductor over the desired range of current, temperature, and magnetic field is a relatively inexpensive means of estimating a conductor's ability to meet a proposed magnet's design requirements. Since such measurements are influenced by the conductor's reaction environment and the strain history experienced by the sample, some care needs to be taken to insure that these conditions are known and are relevant to those of the proposed magnet's.

This paper describes some of the relevant details of the short-sample preparation system, the sample handling and test procedures, and the results of the V-I testing that has thus far been done.

II. System Description

Lindberg "tube" furnaces (75 mm @ 3.8 kW, or 150 mm @ 12.5 kW).were used for controlled atmosphere (Ar) reaction of the wire samples. Oven temperatures were monitored by thermocouples, one of which was used to control the input power in a manner to provide the requested temperature history. The axial profile of input power was adjusted to provide a uniform (5%) region 350 mm long. Samples mounted in a fashion to prevent contact with other samples. Gas purity was maintained by using the highest-purity gas that was available at LBL and by insuring a constant gas-flow against any oven leaks and/or sample outgassing.

All samples were tested while immersed in the magnetic field of a solenoidal magnet built by Cryomagnetics. This magnet was capable of 15 Tesla operation at 4.2 K. The magnet's clear bore was 63 mm and produced less than 1% variation over a range of 60 mm. The magnetic field was usually inferred from the magnet's excitation current and was periodically checked with a Hall-probe mounted within the sample region.

A variable-temperature subsystem fitted snugly within the magnet bore that allowed control of the sample's temperature (T> 1.8 K) by adjustment of either the pumping speed and/or the gas-inlet heater-power. The sample's temperature was measured by a Lakeshore-calibrated resistive temperature sensor that was in thermal contact with the sample. Thermal contact for most of the measurements was obtained by embedding the sensor within a small copper block that was hard-soldered onto a flexible copper foil. This foil was firmly pressed onto the outside of the epoxy-coated sample region with an alumina-loaded heatsink compound and firmly tied into place with nylon twine (to insure good thermal contact during cool-down. The copper foil was insulated on its outside to decrease its sensitivity to the local environmental conditions. The Supercon wire tests utilized two different Carbon-Glass Resistors (CGR's) and had troublesome repeatability. The later IGC sample was tested with a Cernox-style device, with much better results.

Temperature, voltage and current data was sequentially measured as rapidly as the computer/GPIB-controlled instruments could be driven (3.3 Hz)..Raw data was immediately stored in a tab-delimited-ASCII formatted file to allow easy readability by text viewers and easy import into any spreadsheet for later analysis or plotting.

Measurement and calibration was done in the following manner:

1) Temperature: All temperature sensors were monitored by a temperature controller/monitor (Lakeshore-DRC-91CA) that was adjusted to linearly interpolate between the temperature/resistance calibration points supplied by the manufacturer (Lakeshore) of the temperature-sensor. The inferred temperature was periodically checked against the known 1-Atm, liquid-immersion temperatures for LN and LHe. The sensor's magnetic dependence was also determined at 4.2K. All temperatures were post-measurement corrected according to the sensor's 4.2K magnetic dependence. The liquid-immersion absolute system-error was less than 0.2 K at LN and 0.02 K at LHe, after correction for magnetic dependence. The gas-cooled measurements were compared to the liquid immersion results as a cross-check on the two cooling methods.

2) Current: The sample's current was monitored with a 0.200 m-ohm shunt, whose voltage was measured with a digital voltmeter (DVM) (HP-34401A). Periodic comparison with the power supply-indicated current was made to flag any gross miscalibrations. Absolute accuracy depended upon a bi-annual shunt calibration by local specialists and a pre-test/post-test DVM calibration check against a local millivolt standard. The system absolute error was believed to be less than 2%.

3) Voltage: The sample's voltage was delivered by a "low-thermal" shielded-twisted-pair cable to an analog voltmeter for amplification (Keithley-148 @ G = 33000) prior to digitization by a DVM (Keithley-182). The response to a reference voltage source (100 nV, 1000 nV ,and 10000 nV) was recorded at the start of each test-run to check the accuracy of the voltage measuring system and its basic signal-to-noise ratio. This system's absolute error was believed to be less than 5%. The I=0 noise level was usually 50 nV(p-p) with a single-point ground.

III. Short-Sample Preparation

All wire samples were wound onto cylinders as per standard LBL short-sample procedures. Except where otherwise indicated, this meant the following steps were taken:

1) The cylinders (SS-304L, OD = 25 mm, L=300 mm) were pre-insulated with one layer of S-glass cloth. Supercon sample LLL-S1 was an exception where the first glass layer was deleted, in order to determine the extent of any degradation resulting from direct (possibly sintered) contact with the stainless-steel barrel. Standard titanium-aluminum barrels were supplied for comparison purposes. The first tests utilized the standard SS-barrel method in order to increase the chances of reliable data on the first try.

2) Approximately 900 mm of wire was wound onto each barrel-form. Six closely-spaced turns constituted the voltage-sampled region (L=26 mm) and would eventually be positioned at the center of the testing magnet. Two long (200 mm) lead-in legs permitted all splices to occur in low field regions. Saddle regions on the outside of the mounting cylinder provided azimuthal support for the axial lead-splice regions.

3) All regions were tightly held in place before, during, and after reaction by a second layer of S-glass cloth.

4) Both lead-ends were weld-sealed to minimize any loss of tin during baking.

5) The furnace records indicate that the Supercon samples were reacted using a 300 K/hr ramp and a 150 hour flat-top of 657 C. The IGC samples were baked with a 60 K/hr ramp and a 240 hour flat-top of 660 C. All reactions took place in an Ar atmosphere.

6) Cool-down for all samples was "power-off", with an initial cooling rate of 60 K/hr.

7) After reaction, two insulated small diameter voltage tap wires were carefully soldered onto the Nb3Sn conductor approximately 350 mm apart. The wires were positioned relative to the conductor in a bifilar, low-inductance manner and left the sample region as a twisted pair at a location as far as practical from the current leads.

8) After careful cleaning of the splice region, two NbTi leads (each 0.75 mm x 150 mm) were carefully soldered to each Nb_3Sn lead.

9) This system was carefully but tightly wrapped with a third layer of S-glass cloth. The resulting shape was "frozen" into place with a drip-coated layer of Stycast epoxy. The Stycast was thick enough to only penetrate one layer of cloth, thereby leaving the conductor dry and free of epoxy-related strain-degradation.

10) After the epoxy cured, the samples were extremely rugged and ready for mounting onto the current leads.

11) Samples were firmly mounted near the end of the vapor-cooled current supply-leads at a position where the sample-region lies at the solenoid's field maximum and all electrical connections can be made.

12) The sample's flexible NbTi leads were tightly clamped and soldered to their respective vapor-cooled lead-ins.

13) The twisted voltage-tap leads were soldered to corresponding leads from a shielded twisted-pair cable, leading to outside the cryostat.

14) A temperature sensor foil was heat-sunk and tied onto the sample region as described above.

15) After confirming the electrical integrity, a sample was precooled (with LN) and inserted into the cryostat and magnetic field.

IV. MEASUREMENT & ANALYSIS PROCEDURES

The following measuring procedures and philosophies were utilized during the data collection:

1. Measurement started near the lowest stress condition, so we could check the extent to which any sample was damage during testing.
2. Liquid immersion data was taken for all samples, usually at the beginning of testing. Care was taken that steady-state conditions were established.
3. The sample's temperature and voltage-drop were monitored while the current through the sample was ramped until the short-sample voltage limit (10^{-4} V/m) was exceeded.
4. The current was ramped exponentially in a manner to gradually approach the anticipated short-sample current within 10-12 sec.
5. The direction of the current was usually in the tension direction. The compression direction was only checked occasionally (for comparison purposes).
6. If the sample quenched, the current was down-ramped quickly to protect the sample for subsequent testing. If control was maintained, the down-ramp response was measured for an immediate check on the measurement's repeatability.
7. The critical current (I_c) was defined to be the highest current for which the observed voltage gradient was less than $1x10^{-4}$ V/m

V. OBSERVATIONS

The overall status of TPX-relevant short- sample testing at LBL is summarized in Table 1. The listed critical current (I_c) is the average of several measurements. Three of the samples were measured completely. Two of the samples were only tested under liquid immersion conditions: LLL-S1 (for "worst-case" comparison), and LLL-G3 (whose V-tap connection failed upon 2nd cool-down). The TiAl barrel samples were delayed because of their additional construction difficulties.

Most of the voltage data was recorded with the current flowing in the "tension" direction (where the magnetic force is radially outward on the sample region of the coil). Typically raw V-I data exhibited a small amount of noise (200 nV) and some LdI/dt ramp-rate induced voltage (2-3 μV, removed before plotting, Fig.1-4). Current flow in the opposite ("compression") direction always resulted in a slightly smaller critical current, and the V-I data often showed more noise (of the kind associated with wire motion).

TABLE 1: Status of TPX Testing at LBL and Critical-Current (Ic) for T=4.2 K and B=8.0T.

Sample Name	Mfg.	Tube	STATUS	I(A)
1. LLL-G1	Supercon	G-SS	Complete	122.2
2. LLL-G2	Supercon	G-SS	Complete	120.1
3. LLL-G3	Supercon	G-SS	4.2K only	123.0
4. LLL-S1	Supercon	SS	4.2 K only	118.6
5. LLL-T1	Supercon	TiAl	Unreacted	??
6. LLL-T2	Supercon	TiAl	Unreacted	??
7. LLL-T3	Supercon	TiAl	Unreacted	??
8. TPX-A01	IGC	G-SS	Complete	136.3
9. TPX-A02	IGC	G-SS	Ready	??
10. TPX-A03	IGC	G-SS	Ready	??

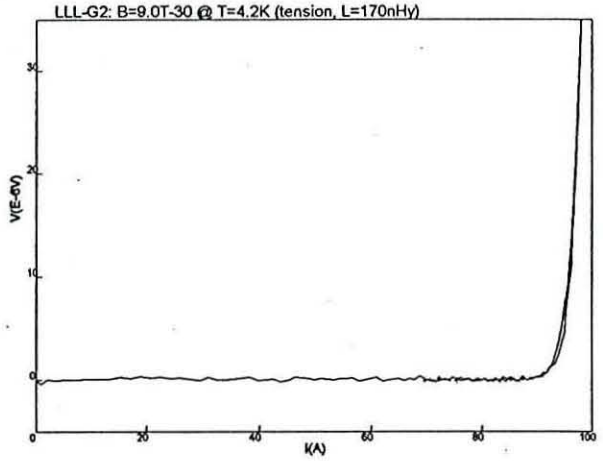

Fig. 1. V-I data: Sample LLL-G2 @ T = 4.2 K, B = 9.0 T, (Tension direction). Up-ramp and partial down-ramp. Voltage was LdI/dt-corrected (L=170 nHy) for display. All sample regions were 35 cm long.

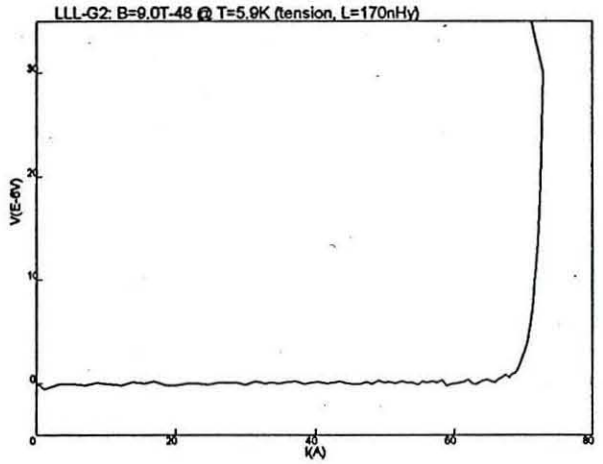

Fig. 2. V-I data: Sample LLL-G2 @ T = 6.0 K, B = 9.0 T, (Tension direction). Up-ramp only (due to quenching at 40 E-6 V). Voltage was LdI/dt-corrected (L=170 nHy).

The IGC sample (TPX-A01) showed higher critical current (see Fig. 5) but a lower superconducting index (N = 20), compared to the Supercon sample (N = 60). This is easily visible in the data (the slower rise of voltage in Fig. 3 & 4, compared with similar conditions for the Supercon sample, Fig. 1 & 2).

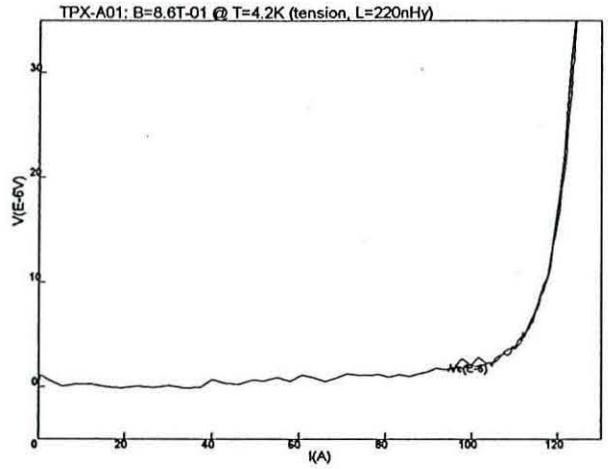

Fig. 3. V-I data: Sample TPX-A01 @ T = 4.2 K, B = 8.6 T, (Tension direction). Up-ramp and partial down-ramp. Voltage was LdI/dt-corrected (L=220 nHy).

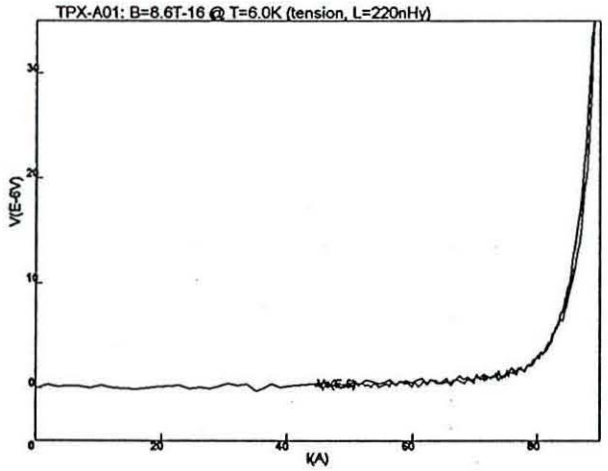

Fig. 4. V-I data: Sample TPX-A01 @ T = 6.0 K, B = 8.6 T, (Tension direction). Up-& down-ramp. Voltage was LdI/dt-corrected (L=220 nHy).

The measured critical currents for all samples tested under LHe-immersion conditions were repeatable and fairly tightly clustered in their respective groups (Fig. 5). Unfortunately, sample-to-sample reproducibility is not yet available foe the IGC wire.

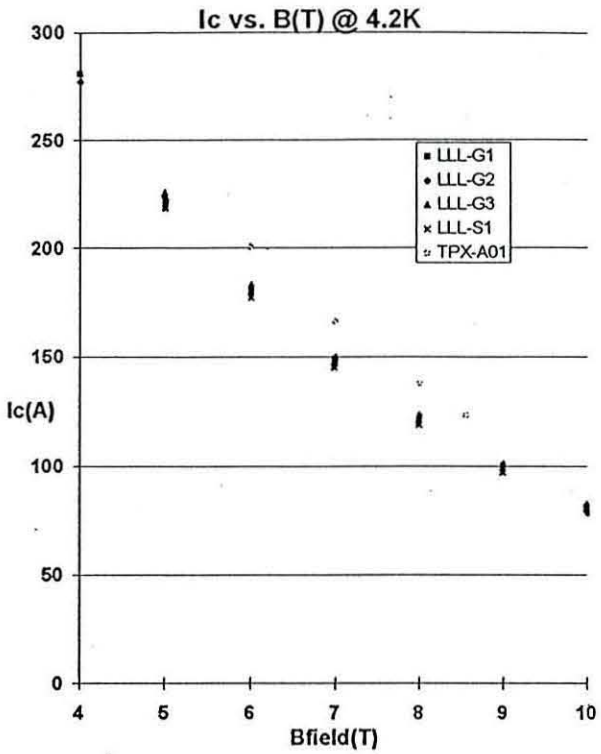

Fig. 5. Critical current vs. Bfield @ T = 4.2 K (LHe immersion). All data taken at E = 1E-4 V/m.

The magnetic and temperature dependence of the critical current for the Supercon samples was tested extensively with samples LLL-G1 (Fig. 6) and LLL-G2 (Fig. 7) because of irregularities that were observed and studied to determine their origins. The magnetic dependence of the temperature sensor was removed according to the dependence observed during the LHe immersion measurements [$T = T_{raw} + 0.013 * B(T)$]. Careful observation of the initial gas-cooled results revealed an unexpected time and pressure-dependent temperature reading from a virgin, but not heat-sunk carbon-glass-resistor (CGR), utilized in the first Supercon measurements. This is believed to have been responsible for some of the data-spread observed at low temperatures and the inability to reproduce the LHe immersion results. Sample LLL-G2 was retested with a new CGR (S2 in Fig. 7) mounted with a substantially improved heat-sinking and thermal insulation. A small systematic improvement was observed, but the time and pressure dependence remained. A retest of sample LLL-G3 was also attempted , but aborted after a voltage-tap opened during its second cool-down. Sample LLL-S1's temperature dependence was not measured.

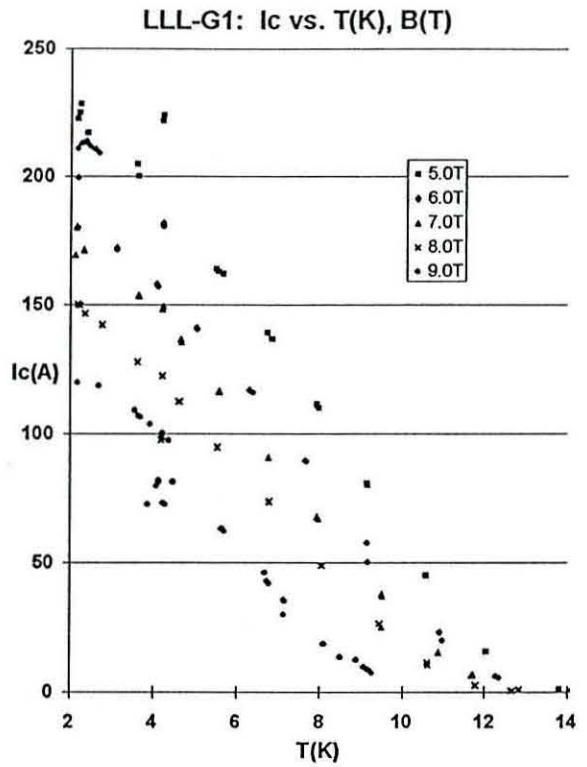

Fig. 6. Supercon sample LLL-G1: Temperature dependence at various magnetic field-strengths.

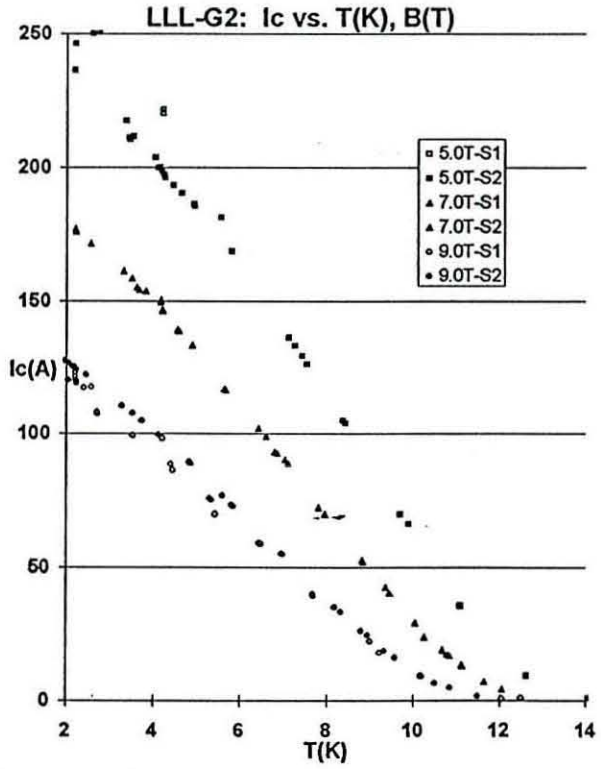

Fig. 7. Supercon sample LLL-G2: Temperature dependence of critical current at B = 5, 7, 9 Tesla, E<= 1E-4 V/m. Sensors S1 & S2 were CGR's, but S2 had significantly better thermal contact to the sample than S1.

A Cernox-style resistor with identical heat-sinking and insulation was used for temperature measurement of the IGC sample TPX-A01 (Fig. 8). It revealed no magnetic dependence at 4.2 K, so no corrections were made to the temperature readings. There was also none of the anomalous pressure or time-dependencies that were observed with the earlier CGR data. Although the gas-cooled critical currents still appear to be lower than the LHe immersion values, they were considerably closer together (Fig. 8). Unfortunately, the maximum attainable field at the time of the measurement was 8.6 T, so, comparable 9.0 T data was unavailable.

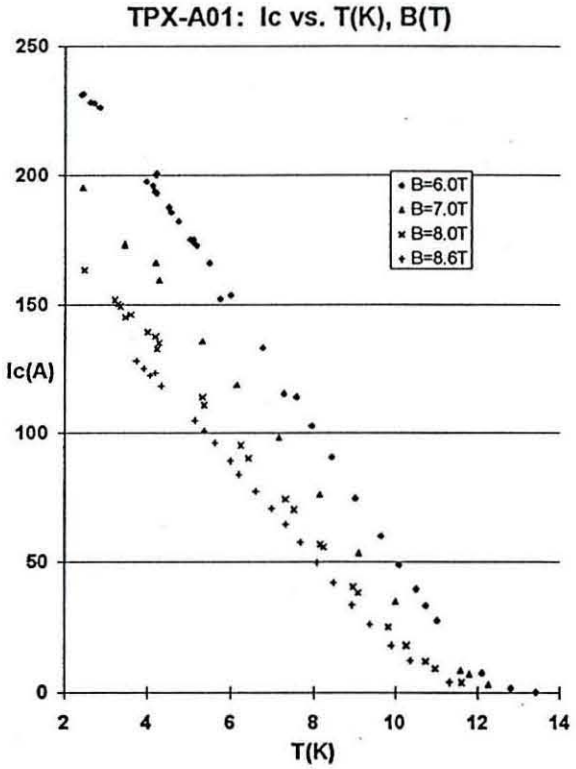

Fig. 8 IGC sample TPX-A01: Temperature dependence of critical current at various magnetic field-strengths.

V. Discussion

Under LHe immersion conditions, the Supercon samples showed good repeatability as long as the wire was separated from the barrel with glass cloth (the LLL-G series in Fig 3). Sample LLL-S1, that was wound directly on the SS-304L barrel, showed a small (2%), but consistent degradation over the entire range of B-fields where it was compared (5-10 T). This data for this graph also included the data for the reverse current direction. Hence, one can conclude that current direction had a negligible influence on the critical current. Close examination of the data reveals 1-2% degradation with the compression direction.

Under gas-cooled conditions, the Supercon samples often showed a current that was lower than expected for the indicated temperature (see Fig. 4). This has been partially traced to the time-dependent pressure-dependence of the Lakeshore CGR that were used. This was an unfortunate result that repeated with a second CGR (both were brand new devices). It not only caused an unexpected delay with the measurements, but it produced results that are excessively pessimistic in regard to the conductor's current-carrying capability, at least at the cold end of the range.

It is not clear how to best correct the data for absolute temperature. One option is to assume that the liquid-immersion data is erroneous. This is highly unlikely in view of the repeatability of the measurements (sample-to-sample and sensor-to-sensor). Another easy option is to assume that the gas-cooled temperature error is roughly constant over the range (as might be caused by a constant temperature difference between the sample and the sensor). One could then correct the data by right-shifting the gas-cooled data until the 4.2 K values agree with the liquid immersion data. However, this introduces the possibility of over-estimating the conductor's capability at elevated temperatures. It is also inconsistent with the lack of improvement observed after heroic efforts were made to improve the thermal contact. A third option is to assume that only the low temperature data had an erroneous temperature (as might be caused by a leak in the sensor's hermetic seal). Assuming the critical temperature to be reasonably accurate, the gas-cooled data could be corrected by rotating it clock-wise in some fashion until the 4.2K data agree. I suspect the last option is the closest to reality, and that such a correction should only be applied to the Supercon data for temperatures below the "kink" observed in the temperature dependence (see Fig. 6). This would still be conservative for an elevated-temperature magnet design.

VI. CONCLUSIONS

Several samples of Nb_3Sn superconducting wire from two different sources were wound onto forms, reacted, and tested at the LBL Short-Sample Test Facility. The anticipated data set was not completed, due to failure of the solenoid magnet. The relative temperature dependence was obtained at several magnetic fields for both kinds of wire; and the relative magnetic dependence was obtained at 4.22 K. Sample-to-sample repeatability was excellent, wherever it was tested. Gas-cooled temperature-dependence repeatability was not good for the early (Supercon) samples, where the sample's temperature inferred from the critical current is at least a degree warmer than the temperature inferred from the temperature sensor. Such data should be used for relative temperature dependencies only, unless they are temperature adjusted to be consistent with the LHe immersion data (believed to be reliable). Subsequent changes to the temperature sensor, considerably reduced the uncertainty in the gas-cooled sample-temperature for the IGC sample (providing the most reliable gas-cooled temperature data in the series). Unfortunately, the magnet failed before the series of measurements was completed.

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