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**Electromechanical Properties of
Superconductors for DOE/OFE Applications**

Final Report

Interagency Agreement DE-AI03-93ER54208

J. W. Ekin and S. L. Bray

DOE/ER/54208--T1

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In many superconductor applications, especially large magnets, the superconductor is required to perform while under the influence of strong mechanical forces. These forces are commonly due to residual fabrication stress, differential thermal contraction of dissimilar materials, and electromagnetic forces generated within an energized magnet coil. Thorough knowledge of a superconductor's electrical performance under the influence of these forces (electromechanical properties) is required for successful magnet engineering. This report presents results of research conducted during the period from August 1993 through March 1997 on the electromechanical properties of superconductors for DOE/OFE fusion applications under interagency agreement DE-AI03-93ER54208. The majority of the research results are contained in three publications that are included in this report. Also, two incremental progress reports, one from Nov. 1994 and one from Nov. 1995, which describe additional results beyond those contained in the publications are also included.

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Executive Summary

Our first publication of research results under this interagency agreement, Characterization of Multifilamentary Nb₃Sn Superconducting Wires for Use in the 45-T Hybrid Magnet, was a collaborative effort with researchers at the National High Magnetic Field Laboratory (NHMFL) and Teledyne Wah Chang. The paper describes the results of characterization studies of candidate Nb₃Sn superconductors for a 45 T hybrid magnet being constructed at NHMFL. When completed, this research magnet will generate the world's highest steady state magnetic field and as a by product, extremely high electromagnetic forces within the coil. Our measurements of the effect of axial tensile strain on the critical current of the candidate conductor at 4 K and 12 T provided the basic empirical data necessary to model the magnet's performance.

A second publication, High Compressive Axial Strain Effect on the Critical Current and Field of Nb₃Sn Superconductor Wire, was motivated by the need to model the electromechanical properties of superconductors used in large stainless-steel reinforced magnets, ITER for example, where the relatively high thermal contraction of the steel can place the superconductor under an axial compressive strain of about -1%. In this study, the axial strain characteristic of a Nb₃Sn superconductor with a high matrix-to-superconductor ratio was tested. The large matrix volume of this wire places the superconductor under an initial *compressive* prestrain of approximately 0.95% at 4 K. Measuring the effect of applied *tensile* strain on the critical current of the precompressed wire provides data over a wide strain range including high compression. A comparison of the resulting data with the Strain Scaling Law developed at NIST showed that this empirical relation can accurately model Nb₃Sn strain effects at high levels of compressive strain.

In the final publication, Tensile Measurements of the Modulus of Elasticity of Nb₃Sn at Room Temperature and 4 K, the stress-strain characteristics of several Nb₃Sn composite conductors were measured to determine the elastic modulus, E, of Nb₃Sn. In modeling Nb₃Sn superconductor systems, E is a key parameter; however, there are large discrepancies in the available data and, prior to this study, there were no published tensile-test measurements of E for Nb₃Sn at low temperature. This study showed that E decreases with decreasing temperature and that its value at 4 K is approximately 65 GPa, compared with 4 K values more than twice this high that have commonly been used for Nb₃Sn. We subsequently used these results for comparative modeling studies of axial and transverse stress effects in Nb₃Sn.

The two incremental progress reports describe additional results of the program including numerous strain characterizations of ITER and TPX candidate conductors; consultations with DOE laboratories and wire manufacturers on strain effects; a comparative study of axial/transverse stress effects in a Nb₃Sn monofilament conductor aimed at extending the Strain Scaling Law from one to three dimensions; and a collaborative study with Intermagnetics General Corp. on the combined effects of reaction-mandrel material and prereaction annealing of Nb₃Sn on critical-current-versus-strain characteristics.

Preprints removed for separate processing.

Tensile Measurements of the Modulus of Elasticity of Nb₃Sn at Room Temperature and 4 K

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Abstract—The critical current of Nb₃Sn superconductors is highly sensitive to strain. Consequently, accurate mechanical modeling of these conductors is necessary to interpret experimental data and to predict conductor performance in applications such as large magnet systems. A key parameter in these models is the modulus of elasticity (*E*, Young's modulus); however, there are large discrepancies in the available data, and there are no published tensile-test data on *E* for Nb₃Sn. Tensile test specimens were prepared from a starting material of Nb tape with 1.4 wt.% ZrO₂ precipitates. Tensile measurements of unreacted Nb and partially reacted Nb-Nb₃Sn tapes were made at room temperature (293 K) and at 4 K. A modulus of elasticity of 65 ± 15 GPa was extrapolated from these measurements for polycrystalline Nb₃Sn at 4 K, and 150 ± 15 GPa at room temperature.

I. INTRODUCTION

Nb₃Sn is presently the most common superconductor used in magnetic-field applications above 10 T; below 10 T, Nb-Ti dominates. Unlike Nb-Ti, however, which is ductile at room temperature and relatively insensitive to strain, Nb₃Sn is brittle and its critical current (*I_c*) at high magnetic fields is extremely sensitive to strain [1]. In magnet applications of composite Nb₃Sn conductors, sources of strain may include coil fabrication, differential thermal expansion between the superconductor and the conductor matrix as well as the magnet structure, and the magnetic forces generated within the energized coil.

The magnetic forces in a solenoid magnet produce orthogonal stress components in the conductor, axial tensile stress and transverse compressive stress. The effects of both stress components on the *I_c* of Nb₃Sn are well documented [1]-[4]. A difficulty arises, however, in comparing their relative effects on *I_c*. Due to experimental limitations, the independent variable in the axial test is strain, while in the transverse test it is stress [3]. To directly compare the two effects, the strain data must be converted to stress, or stress to strain, which requires the modulus of elasticity (*E*, Young's modulus) of Nb₃Sn at 4 K. Also, the modulus of elasticity is important to

Nb₃Sn magnet designers for modeling the mechanical and electrical responses of the conductor to the various sources of stress.

Numerous data have been reported on *E* of Nb₃Sn at various temperatures including 4 K [5]-[13]. The majority of these data are based on ultrasonic measurement techniques applied to single-crystal and polycrystalline specimens. The results of vibrating-reed measurements and static beam deflection measurements of Nb-Nb₃Sn composites have also been reported. Unfortunately, the reported data for *E* of Nb₃Sn at 4 K ranges from 32 GPa to 165 GPa.

In the present study, tensile tests were performed at room temperature (293 K) and 4 K on Nb and Nb-Nb₃Sn tapes. The modulus of elasticity of the Nb₃Sn portion of the composite was then extrapolated from these data at each temperature.

II. EXPERIMENTAL DETAILS

A. Tensile-Test Specimens

Tensile-test specimens of Nb, Nb-1 wt.% Zr, low-carbon steel, and partially reacted Nb-Nb₃Sn (with ZrO₂ precipitates) composites were prepared for this study. In all cases the widths of the specimens are 3.18 ± 0.07 mm. The specimens were carefully cut to their nominal width by simply using a straight edge and razor blade. This technique resulted in a uniform specimen width, along its length, typically within $\pm 1\%$ of the mean. The Nb₃Sn specimens were cut after the reaction heat treatment. The gage lengths of the specimens range from 237 mm to 266 mm. The thickness of the specimens differ for each material. The Nb specimens are approximately 0.075 mm thick, 0.050 mm for the steel, and 0.025 mm for the Nb-1 wt.% Zr as well as the Nb-Nb₃Sn composites.

The 0.025 mm specimens fall into two categories, knurled and smooth. The knurled materials have a mechanically textured surface to promote adhesion of the liquid Sn-10 wt.% Cu coating that is applied to the Nb tape prior to the Nb₃Sn reaction process [14]. The smooth specimens lack this surface texturing. Although the smooth specimens are inherently superior to the knurled specimens for tensile tests, because of their uniform cross sections, the knurled specimens were also tested because they were available in a variety of Nb-to-Nb₃Sn ratios, unlike the smooth specimens. The different composite ratios are achieved by varying the reaction time. Pure Nb₃Sn specimens could be prepared by increasing the reaction time; but, without the ductile Nb core, the specimens would be too brittle to handle. Initially, all of the 0.025 mm specimens had a Sn-10 wt.% Cu surface coating. The coating was removed with a dilute nitric acid solution prior to testing.

The steel specimens were included in the study as control specimens to confirm the accuracy of the measurement method.

B. Measurement Method

The ends of the test specimen are soldered to heavy brass grips, and the specimen is then installed in a rigid cryogenic tensile-test apparatus. The apparatus consists of two stainless steel tubes, an outer stationary tube for force reaction and an inner tube for applying tensile load to the specimen. The brass grips are attached to the load tube and the reaction tube with clevises that allow the grips to pivot in the plane of the specimen, which ensures self alignment and uniform loading. The probe is inserted in a liquid-helium dewar and it is externally connected to a servohydraulic actuator, which provides the tensile load. The actuator is equipped with a linear variable differential transformer (LVDT) for strain measurement and a 1300 N load cell for stress measurement.

The stress-strain characteristic of the specimen is first recorded at room temperature without exceeding its elastic limit. The specimen is then cooled to 4 K in liquid helium, and the stress-strain characteristic is measured again. The modulus of elasticity is calculated from the slope of the stress-strain characteristic. The slope is measured at low strain, typically 0.01% to 0.1%, to ensure pure elasticity.

To measure stress in the specimens, their cross-sectional areas must be determined. Moreover, the area of each component of the composite specimens must be measured to determine E of the Nb_3Sn . After the tensile tests are completed, three samples are cut from the specimen, one from the center and one from each end. The samples are then mounted in epoxy resin and polished for optical microscopic analysis.

The microscopic images are digitized and computer analyzed. The width of each of the three samples is measured, and an average width is calculated. Because many of the specimens are quite thin (0.025 mm), the surface roughness often limits the precision of the thickness measurements. This is particularly true for the composite specimens where the reaction process increases the roughness. To statistically reduce this problem, 90 width measurements of each specimen are made, 30 for each sample, and an average width is calculated. In the case of the composite specimens, both the total thickness and the Nb-core thickness must be measured to determine the component areas, doubling the number of required measurements. Finally, the average width of the specimen is multiplied by the average thickness to determine the cross-sectional area.

The overall uncertainty of the E measurements is dominated by the area measurements and estimated to be $\pm 10\%$. In addition to standard instrument calibrations, including the load cell and the LVDT, a control specimen was prepared from commercial low-carbon steel sheet (0.050 mm thick) and tested at room temperature. The value of E was determined to be 210 GPa, which is comparable to the published values for this material, 208 to 209 GPa.

III. RESULTS

The tensile-test results for the smooth specimens are shown in Fig. 1, which is a plot of E as a function of specimen composition at room temperature (293 K) and 4 K. The data located on the ordinate are from the unreacted Nb-1 wt.% Zr specimens. The remaining data, from the smooth composite specimens, are for a Nb_3Sn content of approximately 46%.

Focusing first on the unreacted Nb-1 wt.% Zr data, Fig. 1 shows a slight increase in E when the specimen is cooled from 293 K (93 GPa) to 4 K (97 GPa). These E values are approximately 11% and 13% lower, respectively, than previously published ultrasonic measurement results for Nb single crystals [15]. Texturing of the Nb grains is the probable source of the reduced E values. Fig. 2 is a photomicrograph of a typical cross section from the composite specimens, showing a central Nb core and Nb_3Sn layers on each side. The appearance of the core is consistent with texturing in the Nb grains. In the tensile tests, the orientation of the applied load is perpendicular to the plane of the figure.

The ultimate purpose of the Nb tensile tests is, of course, to isolate the elastic modulus of the Nb_3Sn portion of the composite specimens by subtracting the Nb-core contribution. The unreacted Nb-1 wt.% Zr specimens, however, are slightly different from the reacted composite's core, which contains ZrO_2 precipitates rather than Zr in solid solution. In either case the effect on E is undoubtedly slight, given the low concentration of additions.

Several specimens of pure Nb were also measured. The pure Nb specimens are approximately three times the

Fig. 1. Modulus of elasticity of smooth specimens at 293 K and 4 K as a function of Nb_3Sn content. Extrapolated values for 100% Nb_3Sn are also shown. The polycrystalline Nb_3Sn contains 1.4 wt.% ZrO_2 precipitates (to limit grain growth and enhance flux pinning).

Fig. 2. Cross-sectional photomicrograph of composite specimen, showing central Nb core and Nb_3Sn outer layers. In the tensile tests, the orientation of the applied load is perpendicular to the plane of the figure.

thickness of the Nb-1 wt.% Zr specimens, which increases the precision of the cross-sectional area measurements. Consistent with the Zr-alloyed specimens, the measurements of pure Nb tape produced relatively low E values, 87 GPa at 293 K and 94 GPa at 4 K, approximately 15% below previously published values [15]. Again, texturing is the likely source of the reduced modulus values, and differences in texturing probably account for the relatively small variation in E between pure Nb and the Nb-1 wt.% Zr. The E values for these textured materials are not indicative of the elastic modulus of nontextured polycrystalline Nb; however, the Nb-1 wt.% Zr data are the required values for solving the problem at hand—isolating E for Nb_3Sn from the composite data.

Turning to the 46% Nb_3Sn composite data, Fig. 1 shows that E is greater for Nb_3Sn than for Nb at room temperature (293 K). At 4 K the opposite occurs; E is lower for the composite, which demonstrates the dramatic softening of Nb_3Sn at 4 K [16]. A linear least-squares fit of the data yields extrapolated Nb_3Sn E values of 150 ± 15 GPa at 293 K and 65 ± 15 GPa at 4 K. The microstructure of these tapes have been studied in detail [17] using transmission electron microscopy (TEM). TEM analysis of the Nb_3Sn layers shows that the fine, equiaxed crystals shown in Fig. 2 are randomly oriented. Consequently, the elastic-modulus values for these tapes are indicative of macroscopically isotropic, polycrystalline Nb_3Sn (with 1.4 wt.% ZrO_2 precipitates).

Fig. 3 is a plot of E as a function of specimen composition at 293 K and 4 K for the knurled specimens. Again, E for the unreacted Nb is slightly higher at 4 K than at 293 K, and for the reacted composites it is significantly lower at 4 K, indicating significant low-temperature softening of the Nb_3Sn .

The data in Fig. 3 are plotted on the same scales as the data in Fig. 1 for comparison. The knurling reduces the measured values of E at both temperatures. This compliance of the knurled specimens is undoubtedly a geometric effect rather than a material property. The knurling creates an undulation along the length of the specimen, which apparently acts like a spring, artificially reducing the measured E value. Consequently, knurled-specimen data are not indicative of intrinsic material properties. The relatively linear relationship between E and Nb_3Sn content for the knurled specimens, however, supports the linear extrapolation applied to the smooth-specimen data in Fig. 1.

IV. DISCUSSION

Ternary additions of Ta, Ti, and Zr to Nb_3Sn have been shown to suppress the martensitic transformation and increase E at 4 K, in comparison with binary Nb_3Sn [10]. The specimens tested in the present study contain 1 wt.% Zr, which is oxidized prior to Nb_3Sn reaction to form ZrO_2 precipitates. These precipitates may suppress the martensitic transformation and, thus, they may have a significant effect on E.

Ternary additions and precipitates are commonly used in Nb_3Sn multifilamentary and tape conductors to improve critical-current density and Nb_3Sn growth kinetics [18]–[20]. Consequently, tensile-test data on these materials are, from an engineering standpoint, as important as data on basic binary Nb_3Sn .

Additional tensile measurements are needed to accurately determine E for pure Nb_3Sn . Ideally, a series of smooth composite specimens of Nb_3Sn and Nb, with different Nb-to- Nb_3Sn ratios like the knurled specimens in the present study, need to be tested to determine a more definitive value of E for pure Nb_3Sn .

Fig. 3. Modulus of elasticity of knurled specimens at 293 K and 4 K as a function of Nb_3Sn content.

V. CONCLUSIONS

There is a broad range of reported values for the elastic modulus of Nb₃Sn at 4 K using various measurement techniques, 32 GPa to 165 GPa. The present data are the first published results on the elastic modulus of Nb₃Sn from tensile tests. These results are qualitatively consistent with most earlier data from ultrasonic, vibrating reed, and static beam deflection measurements, which usually show a low-temperature softening of the elastic modulus. The results of the present study are summarized in Table I.

TABLE I
ELASTIC-MODULUS VALUES FROM TENSILE TESTS OF
POLYCRYSTALLINE Nb₃Sn^a SPECIMENS

Temperature	Elastic Modulus
293 K	150 ± 15 GPa
4 K	65 ± 15 GPa

^aThe Nb₃Sn specimens contain 1.4 wt.% ZrO₂ precipitates and are macroscopically isotropic.

ACKNOWLEDGMENT

The authors thank Christopher G. King of G. E. Medical Systems for preparing the Nb₃Sn specimens and for many valuable discussions.

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Reply to the attention of:

Mail Code 814.05

November 3, 1994

Dr. Marvin Cohen, ER-531
U.S. Dept. of Energy
Washington, DC 20585

Dear Marvin,

Enclosed please find the report on accomplishments for FY'94 and a work statement for FY'95.

Testing is coming along fine and, in particular, the preliminary finite element modeling results for Nb_3Sn are proving to be very interesting. Also, IGC will be sending conductors for us to sort out the reaction mandrel question with a series of electromechanical measurements.

Let me know if you need any more information.

Best wishes,

J. W. Ekin
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Enclosure

LETTER REPORT TO SPONSOR

Electromechanical Properties of Superconductors for DOE/OFE
Applications

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Report Number 814-5-01

November 1994

R. E. Harris, Division Chief
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ELECTROMECHANICAL PROPERTIES OF SUPERCONDUCTORS FOR DOE/OFE APPLICATIONS

Jack Ekin

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DOE/OFE Accomplishment Highlights for FY'94

* A major report combining several years of electromechanical property data was completed and copies were sent to all participants in the DOE/OFE program. The report contains data on conductors that fall in three general categories: Candidate conductors, experimental conductors, and reference conductors. Candidate Nb₃Sn superconductors studied include the USDPC cable strand and an ITER candidate conductor. Experimental superconductors included axial strain measurements on a V₃Ga tape conductor that showed good performance at magnetic fields up to 20 T and three experimental Nb₃Sn conductors containing dispersion hardened copper reinforcement for increased tensile strength. Finally, the reference conductors tested were three different Nb₃Sn conductors from the first and second VAMAS international Nb₃Sn critical-current round robins. Published papers containing key results were also incorporated into the report, including the first measurement of the transverse stress effect in Nb₃Sn, the effect of stress concentration at cable-strand crossovers, and measurements of the electromechanical properties of Nb₃Al.

* Electromechanical property data were also obtained on a Teledyne Wah Chang Nb₃Sn candidate superconductor for ITER. The results showed some variability in the critical mechanical parameters (prestrain and irreversible strain) which helps to explain some of the critical current variability observed in the critical current tests of this conductor. The data also served to cross calibrate Teledyne Wah Chang's measurement apparatus and they used the data as part of their technical exhibit at the recent Applied Superconductivity Conference.

* Measurements were also completed of the transverse stress effect and axial stress effect of a monofilament conductor. These data will serve as the basis for finite element modeling of the three-dimensional stress state in Nb₃Sn. A finite element code has been written and we are starting to analyze the results in an attempt to ultimately generate a *three-dimensional* strain scaling law for use in superconductor magnet engineering.

* Consultation on critical-current testing and the three dimensional strain state in Nb_3Sn strand conductors has also been provided to the Princeton Plasma Physics Lab for design of superconductor magnets.

* Consultation on axial strain effects in Nb_3Sn for use in TPX has also been provided to Lawrence Livermore Lab.

* Consultation on the effects of differential thermal contraction between Nb_3Sn and the reaction mandrel has also been provided for Intermagnetics General Corp. As a result of these discussions, a series of measurements has been planned of Nb_3Sn reacted in both the annealed and unannealed states on both stainless steel and Ti-6-Al-4V reaction mandrels.

Nov. 3, 1995

Dr. Marvin Cohen
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Dear Marvin,

I enjoyed talking with you in Boston the end of August. As we discussed, I am enclosing with this letter the report of accomplishments for FY'95 and a work statement for FY'96. In light of the discussion on funding at Boston, we have scaled back our budget request to about two-thirds of a staff year, \$150 k, from the amount we originally mapped out a couple years ago.

As I mentioned in Boston, testing is coming along fine: we finished four tests on the effect of preannealing the superconductor before winding on the reaction mandrel for IGC, three tests for Mokoto on candidate conductors, and have just been asked by Teledyne Wah Chang to make two tests on their conductors. I am particularly encouraged by some of our recent results on extending the Strain Scaling Law from one to three dimensions and think the deviatoric strain model may prove quite useful in the design of large magnets; we are now working on quantifying the parameters for this design formula.

Let me know if you need any more information.

Best wishes,

Jack

ELECTROMECHANICAL PROPERTIES OF SUPERCONDUCTORS FOR DOE/OFE APPLICATIONS

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DOE/OFE Accomplishment Highlights for FY'95

- * Three Nb₃Sn conductors for the Tokamak Physics Experiment (TPX), two poloidal field (PF) and one toroidal field (TF), were tested. Also, the U.S. ITER candidate conductor, an IGC manufactured Nb₃Sn wire, was measured as part of the second ITER bench mark testing program. Critical current was measured as a function of axial tensile strain at 12 T. From these data the irreversible strain limit and the intrinsic prestrain were determined.
- * In collaboration with Intermagnetics General Corporation (IGC), a series of measurements have been completed on the combined effects of reaction-mandrel material and prereaction annealing on the critical-current vs. strain characteristics of Nb₃Sn conductors. Four specimens of an IGC conductor were tested; two were reacted on stainless steel mandrels and two on Ti-6-Al-4-V mandrels, which are widely used for reacting and testing ITER candidate conductors. The results show that annealing the conductors in an unconstrained state at 200°C for 24 hours, prior to winding the conductor on the reaction mandrel, increases the intrinsic compressive prestrain of the IGC high density conductor from about 0.16% to 0.20%. This increase would be an advantage in large-magnet applications where the conductor may be subjected to high tensile strain from differential thermal contraction and Lorentz force, thus reducing the risk of taking the conductor into the rapidly decreasing tensile side of the J_c vs. intrinsic strain curve.
- * Differential thermal expansion among the components of Nb₃Sn cable-in-conduit conductors, such as those specified in the ITER design, can cause large axial compressive prestrain of the superconducting filaments. Data contradicting the validity of the Strain Scaling Law for large compressive strain were presented at the 1994 Applied Superconductivity Conference by University of Twente researchers. Subsequently, a Nb₃Sn monofilamentary wire, with a compressive prestrain greater than 0.9%, was tested by NIST. Results were in very good agreement with the Strain Scaling Law, and they were presented at the 1995 International Cryogenic Materials Conference along with a related review paper on the Strain Scaling Law.
- * The Strain Scaling Law has been traditionally used by magnet designers to predict the effect of one dimensional (axial tensile) strain on the critical current of high-field superconductors for fusion applications. Progress is now being made in extending the Strain Scaling Law from one dimension to three dimensions in order to predict the composite effect of multidimensional stresses within the windings of large magnet systems such as ITER. A simple conductor geometry, a multilayer Nb₃Sn tape, was modeled. Three Nb₃Sn tape conductors (two custom-made non-knurled tape conductors from General Electric (GE) and one tape conductor from IGC) were also tested under uniaxial tension for eventual comparison with the transverse compression data. A

preliminary transverse stress test was also run on the GE tape conductor. For the transverse-compression case, the stress model predicts an initial increase in critical current, due to a reduction in deviatoric strain, when transverse compressive stress is applied. The transverse test showed this prediction to be at least qualitatively true. This has also been corroborated recently by an independent test at the University of Twente. A quantitative comparison with the deviatoric model is now under way.